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NOTICES.—All communications relating to editorial matter should be addressed to the Editor, who will be pleased to consider articles or contributions dealing with modern chemical developments or suggestions bearing upon the advancement of the chemical industry in this country. Communications relating to advertisements or general matters should be addressed to the Manager.

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The Past Twelve Months

IN this issue we publish a series of articles reviewing the year so far as the interests and activities of the various branches of industrial and engineering chemistry are concerned. The authors of these articles speak with knowledge and authority, and in their desire to do justice to their subjects they have not overlooked many of the minor developments which have taken place and which, in no small degree, have exerted a considerable influence in the larger field. Mr. J. Davidson Pratt, as general manager of the Association of British Chemical Manufacturers, is in a unique position to deal with such matters as come within the scope of his review. Then there are Dr. E. B. Maxted, who writes on the nitrogen industry; Colonel G. P. Pollitt, on the progress made by Imperial Chemical Industries; Mr. P. Parrish, who is ever welcomed for his rich store of information on conditions in the heavy chemical industry; and Mr. H. W. Cremer, a new contributor to these pages, with his article on chemical engineering progress. Other features deal with problems of national importance which have fallen within the scope of the Department

of Scientific and Industrial Research; the work of the Association of British Chemical Manufacturers and its affiliated organisations; the British synthetic fertiliser industry; the aliphatic solvent industry, and the dyestuffs situation.

The year 1931, like its predecessor, has been a depressing one. Depression has slowly crept into nearly all industries, so much so that its effect has been reflected in the slowing down or complete abandonment of many development schemes as well as of research work. Troubles thickened during the summer when a financial crisis came with great suddenness in Germany, which is predominantly a chemical nation, whose operations are closely entwined with the chemical industry of Britain and Europe generally. Then came President Hoover's proposal concerning the suspension of war debt payments for one year, but this gesture did not receive the immediate response which was expected. The events which followed need not be related in detail to remind us of the anxious times through which we passed and which culminated in the formation of a National Government and subsequently, the suspension of the Gold Standard. The General Election confirmed the National Government in power with a mandate to redress the adverse balance of trade. The task of the Government will not be a light one, but it should be accomplished if there is determination in tackling the many problems for which solutions have still to be found.

The outstanding event of the year was the British Chemical Plant Exhibition which was held in London in July. This exhibition was staged to show the great progress which had been made in chemical engineering since 1926, when the previous plant exhibition was held. Much of the plant exhibited was publicly shown for the first time, and it was obvious that the intervening period had seen many progressive developments, the trend of which clearly indicated that the prosperity of Britain was becoming more and more dependent upon an underlying foundation of chemistry. During the same week the Society of Chemical Industry celebrated their Jubilee in London. Many visitors came from overseas and Professor G. T. Morgan was elected to the presidency of the Society in succession to Sir Harry McGowan.

On the development side the interests of the industry have chiefly centred around the possibility of producing oil from coal by the hydrogenation process. Confident opinions have for some time been expressed that under proper conditions, and with adequate support, the conversion of coal into oil might become an important new national industry. At Billingham, Imperial Chemical Industries have already erected a plant which is capable of treating 15 tons of bitu-

minous coal a day, of which 60 per cent. is recovered as refined petrol. Rapid advance in the technique of this problem has most probably been one of the results of the formation of the International Hydrogenation Company through whom patents and knowledge were pooled. Still we must not overlook the fact that the huge chemical works at Billingham are possessed of a unique experience in high pressure work, and as a centre of research in industrial chemistry and chemical engineering it is one of our greatest national possessions.

With the continuance of the Dyestuffs Act for yet another year and the number of dyes we cannot produce in this country gradually diminishing; with the British synthetic fertiliser industry more firmly entrenched at the end of this year than ever it has been; with the increasing demand on an industrial scale for many new solvents; and with all the recent progress which has been made in the production of heavy chemicals as well as fine pharmaceutical products; there are certainly great hopes for the future. Nearly all industries are now dependent upon the use of chemicals in small or large quantities, so that the British chemical industry as a whole has every chance to make headway. It should be quick to seize each opportunity that is offered for making and selling new products in the homeland as well as overseas.

Unsuspected Arsenical Poisoning

At the adjourned inquest held in Liverpool concerning the death of James Parry, furnaceman, and Thomas Lowe, labourer, both of whom were employed by a firm of tin smelters at Bootle, the Coroner, Mr. G. C. Mort, emphasised the importance of notifying cases of suspected arsenical poisoning. These men were employed at the largest tin smelting works in the world, that of Williams, Harvey and Co., Ltd. Evidence was forthcoming that Lowe, Parry and four other men were engaged in removing ashes from one of the refinery furnaces, and that their illness started with weakness in the legs, shivering and violent pains in the stomach. Ever so far back as 1928 there were several cases of illness among the employees, but these cases were thought to be due to terpene poisoning derived from the sawdust used in the treatment of the residual dross remaining after most of the tin had been extracted from the ore. The men, whilst engaged in raking over the residue, were equipped with respirators, but as the symptoms of poisoning persisted it was thought that some of the dust was passing through the respirators. In consequence, improvements in the respirators were made. No further cases occurred until January, 1931, when two men were affected and the Company, on the advice of their medical officer, called in an expert who diagnosed the systems as arsenical poisoning. Certain additional recommendations were then carried out, but in May there were two more cases of illness and very strict regulations were issued regarding the use of respirators. The climax came in November when the six men, referred to above, were taken seriously ill, and the poisoning was found to be due to arseniuretted hydrogen—a gas produced by the action of water on the baked residue of sawdust and tin dross, and one, moreover, for which there was no effective respirator available. The employers did everything in their power to protect their employees,

but the Coroner emphasised the importance of immediate notification to the District Inspector of Factories in cases such as this. This was a new unsuspected cause of poisoning which should be brought to the notice of all concerned in the chemical and metallurgical industries.

A Postponed Conference

THE American Institute of Chemical Engineers, recognising "the possibility that disturbed economic conditions may well persist another year," have postponed their joint meeting with the British Institution of Chemical Engineers, arranged to be held in London in June, 1932. According to the correspondence that has passed between the two organisations, this motion was unanimously carried at a special meeting of the Council of the American Institute held on October 30 for the purpose of discussing plans for their reunion with the British Institution. The Council of the Institution of Chemical Engineers have acknowledged the sentiments actuating the American proposal, which they regard as a friendly gesture, recognising that it has probably been dictated as much by the consideration of conditions in this country as by doubt concerning the number of American chemical engineers who would be able to attend the conference. It is, nevertheless, regrettable that the American Institute have been obliged to make this postponement, for in times of economic difficulty there is still much to be said for conferences of an international character, which, moreover, are, directly or indirectly, concerned with industry. With any appreciable improvement in general conditions early in the New Year, it is to be hoped that the American Institute will be in the happy position to review this decision, for even then it should be possible to complete the necessary arrangements which the British Institution have already drafted for welcoming their American cousins, with free interchange of practical data and theories. In any case, the visit is merely postponed and not abandoned.

Brotherton Research Scholarship

THE late Baron Brotherton, of Wakefield, bequeathed a sum of £20,000 to be devoted to charitable and educational purposes in the city of Leeds, and of this sum the Executors in consultation with the Lord Mayor of Leeds have allocated £2,500 for the foundation of a Research Scholarship in some branch of industrial science, preferably industrial chemistry.

The Scholarship will be known as "The Brotherton Research Scholarship," and will in general be of the value of not more than £100. It will be awarded for a period of one year for an approved course of research at a British University and be open to men and women who have attended a Leeds school for a period of at least three years immediately prior to entering a university. Candidates must have graduated in Honours and have shown special capacity to profit by a period of research. Students who have not already graduated but who are taking their final honours degree examination in the year of award may submit applications.

The Scholarship will be awarded by a Committee to be known as the Brotherton Research Scholarship Committee, consisting of the Lord Mayor of Leeds, the Vice-Chancellor and two other representatives of the University of Leeds, the Chairman and Vice-Chairman of the Leeds Education Committee, the Director of Education for Leeds, and Mr. C. F. Ratcliffe Brotherton. The Committee will take into account the University records of the candidates and the course of research proposed to be followed, and the award shall be subject to the Committee's approval of the proposed course and of the University at which it is to be undertaken.



Photo]

[E. O. Hoppe

BILLINGHAM: THE PLANT FOR CONVERTING WATER GAS TO HYDROGEN.

A Review of British Chemical Industry in 1931

By J. Davidson Pratt, O.B.E., M.A., B.Sc., F.I.C.

As general manager of the Association of British Chemical Manufacturers, Mr. Davidson Pratt has exceptional facilities for accurately surveying the conditions of the past year so far as the British chemical industry is concerned.

It was thought that 1930 was a bad year but 1931 has been worse. An examination of the Index of Production issued by the Board of Trade shows that, for the chemical group of industries, production reached a maximum of 115.8 in 1929 (1924 being taken as 100). The quarterly figures for 1930 were 106.3, 101.3, 105.5 and 94.6, with an average of 102.2 for the whole year. For the first three quarters of 1931 the figures were 101.4, 96.1 and 91.1. The figure for the last quarter should, however, show an appreciable increase owing to the revival of trade which resulted from our departure from the gold standard towards the end of September, with its consequent competitive advantages in the home and export markets. It is interesting to note that the index figures for the chemical industry follow fairly closely the average index figures for British manufacturing industry generally; these latter are as follows:—1929—115.5; 1930—106.3; 1931, first quarter—98.0, second quarter 95.0, third quarter 93.2. Thus the condition of the chemical industry is a good index of our general industrial prosperity. This follows naturally from the fact that the chemical industry is a supplier of raw materials for every industrial activity.

The employment statistics issued by the Ministry of Labour also make unsatisfactory reading, though here again the chemical industry approximates closely to the average for industry as a whole. At the end of 1930, the estimated number of insured persons between the ages of 16 and 64 in the chemical group of industries was 102,930, while the percentage unemployed had nearly reached 18. The latest figures

for insured persons in the industry are 98,460 and the percentage unemployed is between 18 and 19.

Foreign Trade

The export and import data give cause for considerable anxiety. In 1930, the value of the exports for the group "chemicals, drugs, dyes and colours," as defined in the Board of Trade list was just on £22,000,000, a drop of some £4½ million on 1929. The figures to the end of October, 1931, indicate that there will probably be a further drop of nearly 25 per cent. this year. There is a falling off in the value of the chemicals exported under most of the sub-headings of this group. The decreases are particularly heavy for sulphate of ammonia, coal tar products, sodium compounds and painters' colours and materials. The fall in price levels has been responsible for a considerable part of this reduction and, if judged by quantity, the reduction is not so great as it is on a value basis. The 1931 exports for sulphate of ammonia are likely to be only half the value of those in 1930, though the decrease is not so great on a tonnage basis. The value of the coal tar product exports has also dropped to about half, mainly due to creosote oil, but here again the value has dropped more than the quantity. It is, however, gratifying to note an increase of about 13 per cent. in the value of the synthetic dye exports.

Imports have also dropped but only by about 7 per cent., the 1930 value being £13½ million, which represented a decrease of nearly £3½ million on 1929. There have been

reduced imports on a value basis in the case of acetic acid, coal tar products, potassium nitrate, cream of tartar, zinc oxide, drugs, medicines and medicinal preparations, and painters' colours and materials. On the other hand, there have been increases in a number of cases of which the following are important:—Tartaric acid and tartrates, glycerine (distilled), potassium compounds, sodium nitrate and miscellaneous chemical manufactures. Certain of these items definitely fall in the category of abnormal imports, obviously made with the object of forestalling a Government tariff. In regard to quite a number of products, dumping has been taking place more or less systematically throughout the year, and our manufacturers have had to contend with increased competition of this type from Soviet Russia which has put various chemicals on the British market at prices far below anything which is economic here. Russian competition has also been encountered in the Far East markets where they are prepared to undersell any price which the British trader may quote. The menace from this quarter requires to be carefully watched as it may grow with the progress of the Five Year Plan.

An analysis of our overseas trade for the first nine months of 1931 at the 1924 values indicates that, compared with 1924 as 100, the value of the exports for 1931 is 73.2 and the quantity 91.6; for imports, the value is 85.7 and the quantity 99.6. This shows that, while imports have remained fairly steady in quantity, there has been a 10 per cent. drop in the quantity of the exports. Value levels have dropped some 15 to 20 per cent.

The Sulphuric Acid Barometer

The following figures in regard to the production of sulphuric acid in the United Kingdom and Ireland are of interest as an indication of the general state of British industry during the last three years:—

Output of Sulphuric Acid and Plant in Operation.

Year.	Tons 100% H ₂ SO ₄ .	Percentage of Capacity.
1928—last 6 months	471,000	67.3
1929—first 6 months	488,000	71.3
1929—last 6 months	479,000	70.3
1930—first 6 months	463,000	68.3
1930—last 6 months	387,000	58.8
1931—first 6 months	371,000	58.7
1931—July, Aug., Sept.	136,500	43.8

It will be noted that whereas during the first six months of the present year the output recorded was the lowest for many years past, the third quarter shows a reduction of over 25 per cent. on this previous low record. This grave state of affairs is in the main due to the decrease in the consumption of sulphuric acid by the sulphate of ammonia industry, caused by the very serious fall in the price of this commodity. Since September the price of sulphate of ammonia has recovered to some extent, but there does not yet seem to be any clear indication that this recovery in price is accompanied by an increase in the manufacture of by-product sulphate.

In the spring of this year determined attempts were made by certain foreign manufacturers to market in the United Kingdom large quantities of sulphuric acid obtained as a by-product, under conditions which could only be described as dumping. Though our acid makers were able to resist these attacks, the danger of dumping still persists and can only be removed when adequate protection is afforded to the British manufacturer. Otherwise the future of this basic industry on which a large number of industries depend for an essential raw material will be seriously jeopardised.

As regards raw materials, figures for comparison are only available for the first six months of 1931, and are contained in the following tables:—

Total Acid Made and Proportions of Raw Material Used.

Year.	Total Acid, including Oleum, in tons 100% H ₂ SO ₄ .	Im- ported Pyrites	Percentage made from Do- mestic Pyrites	Spent Oxide	Sulphur	Zinc ores.
1929 ..	967,000	47.54	0.81	24.62	17.71	9.32
1930 ..	850,000	50.16	0.61	25.33	13.98	9.92
First six months of 1931	371,000	51.07	0.73	24.85	13.54	9.21

Output of Sulphuric Acid and Consumption of Raw Materials in Tons.

Year.	Acid & Oleum.	Net Acid.	100% H ₂ SO ₄ Oleum.	Pyrites.	Spent Oxide.	Sulphur & H ₂ S. ores.	Zinc ores.
1929	967,000	808,500	158,500	363,500	174,500	52,700	115,600
1930	850,000	696,000	154,000	336,500	155,800	36,900	109,100
First six months of 1931	371,000	305,000	66,000	153,000	67,000	15,400	39,900

The chief item of interest is the decrease in the consumption of Sicilian sulphur, which may be attributed in the main to a very considerable increase in the price of this material. It is extremely doubtful if sulphur will continue to be used in Great Britain as a raw material for sulphuric acid manufacture, unless it can be obtained on more advantageous terms than those at present maintaining. The percentage of acid manufactured from pyrites continues to increase.

The spent oxide position has been very directly affected by the adverse trade conditions, and in particular by the sulphate of ammonia position referred to above. The result of these factors is that at the present time there is some accumulation of stocks of spent oxide at the gas works, but the position is believed to be only a transitory one, which would automatically cease to exist should trade conditions improve.

The following figures extracted from the Annual Report of the Chief Inspector under the Alkali Act, give the number of works (as distinct from firms) licensed to manufacture sulphuric acid (in the United Kingdom and Ireland) during the years named as follows:—

Year.	Number of Works.
1915	171
1920	156
1925	134
1930	118

This table shows very clearly the gradual concentration of manufacture in larger units with accompanying rationalisation which has taken place during recent years. Indeed, during the current year no less than ten firms, formerly manufacturing sulphuric acid have ceased to do so, by agreement with other manufacturers. With regard to sale price, in spite of the increased cost of raw material referred to above, the general trend during the year was downward.

In view of the fact that 75 per cent. of the raw material necessary for the manufacture of sulphuric acid has to be imported, an entirely new set of circumstances arises since the departure of Great Britain from the gold standard on September 21. What the effect of this will be, it is too early even to conjecture, but obviously many possibilities may arise.

General Chemicals

The position in regard to the basic heavy chemicals during the year 1931 may be summed up in a few words. There has been a steady decrease due to the widespread depression in most branches of industry until we left the gold standard; since then, there has been a marked improvement. This applies to the export as well as the home trade. Generally speaking, the excess of productive capacity over consumption has tended to increase, with the result that price levels have tended to fall. Since our departure from the gold standard prices have, generally speaking, remained fairly constant, and manufacturers have not attempted to exploit the position. Where imported raw materials have to be used, the British manufacturer has naturally been forced to raise the price to recoup himself for the resultant increase in his prime costs.

In 1930, the exports of general chemicals (that is, chemicals other than fertilisers, medicines, dyes, coal tar products, and painters' materials) were valued at just over £9,000,000, but are likely to be between 15 and 20 per cent. lower in 1931. The imports for 1930 were valued at £5,000,000. For 1931 they are practically as heavy though a decrease should have been expected in view of the general depression. This position may be explained by the fact that there have been heavy abnormal imports in a number of cases, such as ammonium chloride, boric acid, tartaric acid and tartrates, and citric acid, which can be attributed only to an attempt by the foreigner to forestall the imposition of a general tariff.

Fertilisers

The world position in regard to nitrogenous fertilisers has been reviewed in the annual report of the British Sulphate

of Ammonia Federation, which will be issued in January. In addition to the general depression in British agriculture which has steadily become worse, British producers of sulphate of ammonia have been adversely affected by the failure of the International Nitrogen Convention to come to a new agreement. As a result a great many European countries have imposed a system of import prohibition and licence on nitrogenous fertilisers to protect their own manufactures. Another outcome of the situation was that Chile resumed open market sales such as existed before the first international agreement was made two years ago. This country took no action to protect its sulphate makers so that we have become a dumping ground for Chile nitrate which cannot find its intended market in other European countries. The imports of Chile nitrate to the end of October were some 250,000 tons in advance of the corresponding period of 1930, an increase of some 35 per cent., and further abnormal consignments are known to have been imported in November. It is hoped that the Government will take appropriate action under the Abnormal Importation (Customs Duties) Act. The price of sulphate of ammonia has also been affected; it dropped by about 30 per cent. in August and it has since remained at a low level. A considerable number of gas works have found that they were losing money on their sulphate at this very low price and have ceased production, but even now capacity is still far in excess of requirements. Exports of sulphate of ammonia in 1930 were roughly 558,000 tons, valued at £4,400,000. On the basis of the first ten months of 1931, the tonnage exported is likely to be down by 30 per cent. and the value by over 40 per cent., the main reductions being in the case of Spain and the Canaries, China and Japan.

The position of the British superphosphate manufacturer has also been critical. Not only has the serious financial position of the farmer hit the superphosphate industry but it has also had to contend with foreign material which has been imported in even larger quantity than in 1930 when over 100,000 tons, valued at £267,000, came into this market. The quantity of superphosphate produced in Great Britain and Ireland during the last fertiliser year, ending June 30, 1931, was 506,000 tons, as compared with 575,600 tons in the previous year, a reduction of about 12½ per cent. Exports are small, being only 18,000 tons in 1930, valued at £64,000; these figures will be about halved in 1931.

The future welfare of our fertiliser industry is dependent on the action taken by the Government to stimulate British agriculture and to remove it from the slough of despond in which it has languished for the past few years.

Coal Tar Products

During the first eight months business in coal tar products generally was dull and the prices in most cases showed a decrease as compared with the end of 1930. When this country went off the gold standard, business became temporarily brisker and there was an improvement in prices.

Exports of creosote show a serious drop. This year's figures to the end of October are only about 50 per cent. of the 1930 figures for the same period, both as regards quantity and value, so that whereas 27,000,000 gallons were exported in 1930, only half this amount is likely to be shipped in 1931. Pitch, on the other hand, shows a more favourable result. The concerted efforts that have been made to stabilise the market in this product have been attended with some success. The price during the year advanced gradually by about 10 per cent. until our departure from the gold standard; since then, there has been a considerable improvement. Unsold stocks are much reduced and the forward position is firm. During 1931 the demand for road tar was again very good and exports to the Continent showed a considerable advance. Prices were roughly on the same level as during the previous season. Down to about the end of August tar spirits shared the general depression and values were affected by the various reductions in the price of petrol. Recently an increased demand has set in and prices have improved. There is a shortage of toluol and the price is now 3d. higher than the figure ruling at the end of 1930. Carbolie and cresylic acids dropped in price until September, but since then the position has improved. Pyridine bases have been quiet throughout the year and production has fallen owing to the unremunerative prices obtainable. There has recently been an increase in the demand and values have advanced.

In 1930, exports of coal tar products were valued at £1,154,000. The 1931 figures are likely to be only half this. This heavy reduction is due mainly to creosote oil as has already been mentioned, but all other tar products have been affected as well. The imports which, in 1930, were valued at £300,000 have dropped by some 30 per cent.

There have been no developments of outstanding importance during the year but work is continually in progress, largely by standardisation, to place on the market products of higher and more uniform quality.

Wood Distillation

As in many other British industries, the past year has been mainly one of depression for the British wood distillation industry. During the first six months trade was fair, but the demand gradually faded away, resulting in almost complete stagnation until our departure from the gold standard when an immediate increase in demand took place. At the present time, the prospects are much better than they have been for some years, but this is due entirely to the premium our manufacturers are obtaining through the exchange rates of their main foreign competitors. The imports of wood distillation products usually come from nearly all countries; during 1931, Soviet Russia has joined the rest of the world in sending us her charcoal.

At the time of writing, it is not possible to give production statistics for 1931. There can be no doubt that the final figures will show a considerable reduction on 1930 and previous years. This reduction was brought about by several causes. The first may be accounted for by world-wide imports at uneconomic prices. The second is due to the commercial production in this country of synthetic materials in the form of methyl alcohol, acetic acid and acetone. Throughout its entire development, the wood distillation industry has suffered reverses owing to the manufacture of substitutes for its normal by-products. To-day, it is suffering through the success of modern chemical development and the demand of industry for more and more material which wood distillation could not provide in sufficient quantities as required. The production of acetic acid requires large quantities of wood to secure the acetate of lime as the raw material. At the same time, all the attendant products, both primary, such as charcoal, and secondary, such as wood spirit, tar, etc., have to be worked up and sold. If no demand exists for the by-products, the production of acetic acid declines. The same applies to acetone. It was, therefore, obvious that with the rapidly increasing demand for certain of the products of wood distillation which the industry could not regularly supply, new sources of production must be sought. This demand has been met by the manufacturer of acetic acid by two different methods, the production of acetone by fermentation and the supply of synthetic methyl alcohol.

In the meantime, the industry has not been entirely idle in investigating new methods of production. The main handicap in the form of bulk quantities of wood providing only small amounts of by-products of value still remains, and although there seems at present no method of overcoming this difficulty, nevertheless success has attended recent investigations. New methods are providing larger yields of by-products suitable for the requirements of certain industries, such as the paint and lacquer trades. One well known Scotch firm has adopted the Suida process for the production of strong acetic acid direct from pyroligneous acid. It is too early yet to say whether their example will be followed by other manufacturers, but any process which will strengthen the position of British wood distillers in their competition with imported and synthetic materials will receive most careful consideration by the industry.

Dyestuffs

So far no figures are available for the production of dyestuffs and intermediates in Great Britain during the current year, but it is probable that output will be on the 1930 level in spite of the great depression in trade generally during the first nine months of the year. Since the fall in the value of the £ there has been an increased demand for colours, particularly for the heavy woollen trades. With the altered circumstances which have also had a beneficial effect on other textile trades as well, there is reason to believe that the dyestuff industry will be called upon to supply increasing quantities of dyes. The home trade which calls for the best class

of materials and colours, has been fairly well maintained throughout the year.

The total amount of dyestuffs and intermediates manufactured during 1930 was 42,590,000 lbs. showing a decrease as compared with 1929 of approximately 13,000,000 lbs. This was due to the great depression in the dyeing and printing trades, both for the home and foreign markets, which created a position of stagnation hitherto unknown to these British industries. This is reflected in the figures for the production of indigo which showed a falling off of nearly 5½ million lbs., due to the lessened demand. In spite of the state of trade, vat colours showed a production of 2,518,000 lbs. in 1930 as compared with 2,659,000 lbs. for 1929; this is a gratifying feature and is due to the introduction of several additions to this series of fast colours.

The dye industry has also been affected by a number of factors operating outside the country, such as the difficulties in India, the uncertainty in the Chinese and Eastern markets, and the political unrest in some South American countries. These have all contributed to lessen the demand for dyed and printed goods. At the same time, it is gratifying to note that there is likely to be an increase of some 13 per cent. in the value of dyes exported this year. The figure for 1930 was just over £1,000,000. Imports have remained fairly steady in value and are likely to be just over £1,000,000 for 1931. There were abnormally heavy importations under licence of dyes not made here in September, October and November, due, apparently, to the fear of British users and foreign makers that a tariff was to be imposed. The continuance of the Dyestuffs Act for another year will remove this fear and it is likely that imports will drop, especially as our depreciated currency will make such dyes a somewhat expensive luxury, except in the limited cases in which they are really essential. This will give the British maker an opportunity of coming into his own, as it has long been felt that many of the foreign colours which are imported are not really essential and could quite well be substituted by British-made products.

There has been no important alteration in the prices of dyewares during 1931 and British prices still appear to compare favourably with those ruling abroad. That this is so is confirmed by the fact that there have been practically no licences granted this year on price grounds, though such cases were dealt with on the basis that the British maker had to meet the fair foreign price. As already indicated, important additions have been made by British makers to the range of fast colours both for wool and cotton, and there is every reason to believe that the existing gaps will be filled in the course of time. An outstanding feature has been the discovery and introduction by British manufacturers of the first green of the Azoic series which, when coupled with the appropriate bases, yields bright green and blue shades with excellent properties.

Fine Chemicals

The production statistics for fine chemicals collected by the Association of British Chemical Manufacturers show that in 1930 the volume of output, as judged by weight, increased by some 15 per cent. over the 1929 level, though the value remained practically unchanged at slightly over £5,000,000, owing to the fall in price levels. The import data for key industry chemicals also make satisfactory reading. The value of fine chemicals entering from foreign countries in 1930 was £525,000, as compared with £777,000 in 1929, while the fine chemicals from Empire countries—these entered on the Empire preference basis, that is, free of duty—were valued at £298,000 and £563,000 respectively. Thus, the total fine chemical imports for 1929 and 1930 were £1,340,000 and £823,000 respectively, a drop of nearly 40 per cent. While part of the decrease can be attributed to the fall in price levels and the reduced demands of British industry, by far the greater share has resulted from the steady progress of new manufacturing enterprises in this country, such as acetic acid and butyl alcohol.

For 1931, the production figures will certainly show a decline owing to the depression in consuming industries. As with other chemical manufactures, the suspension of the gold standard has given a much desired stimulus to buying, both for the home and export markets, and many buyers both here and abroad are now finding it advantageous to purchase

their requirements in this country. Prior to that, trade at home suffered from a sort of sleepy sickness due to the unwillingness of buyers to issue stock orders, and their plan of proceeding on a hand-to-mouth principle. The poor summer has adversely affected the trade in photographic chemicals. Export trade has also been under a cloud for a variety of reasons, apart from the inability of foreign purchasers to pay for more than an irreducible minimum of requirements. The most serious influences have been the boycott in India which could be settled by a firm Government policy, the very low value of silver, and internal troubles in China, and the rubber depression in the Strait Settlements. Accordingly on both accounts the volume of trade secured by manufacturers has been greatly below normal. The total exports of fine chemicals cannot be ascertained from the published statistics, but the figures for drugs, medicines, and medicinal preparations are significant. In 1930, the exports under this head were valued at £2,836,000, a drop of about 12½ per cent. on 1929. For 1931 there will be a further drop of 5 per cent.

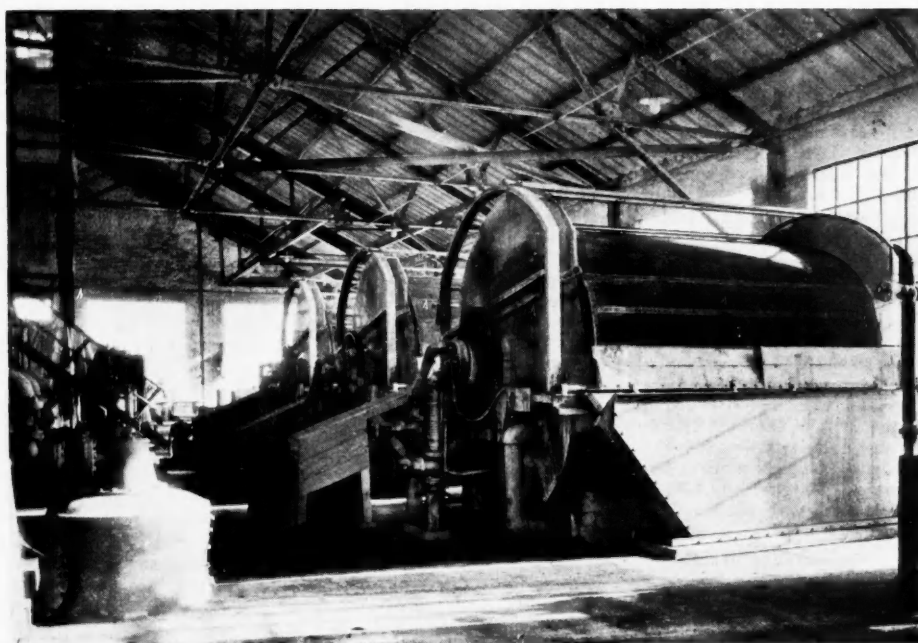
New Developments

There are a number of new developments of interest. British production on a large scale of lactic acid B.P. and of phenolphthalein has been commenced. There has been a further application of tin compounds, such as methyl stannic iodide, tin sulphur iodide, and methyl tin lactate in medicine; the commercial production of salts of Kurchine, the alkaloid of Indian Kurchi Bark, for the treatment of dysentery; the perfection of a starch-free food for diabetics from Carrageen (Irish moss); the large scale manufacture of pure crystalline vitamin D (Calciferol) recently discovered by Bourdillon and his colleagues at the National Institute for Medical Research at Hampstead; and the production of a highly concentrated preparation of vitamin A, of a standardised liquid preparation of ergot and of ergotoxine ethanesulphonate. An increasing number of dyestuffs are being prepared in a highly purified condition for use in medicine, among which may be mentioned Brilliant Green, Fluorescein, Indigo Carmine, and Trypan Blue. A feature of special importance has been the appearance of Empire Bromine from the Dead Sea area, which has rendered bromine makers independent of the Continental bromine ring. The foreign bromide makers at once tried to stop this by intensive dumping of bromides in this market at prices far below those ruling in the country of origin. This competition is still going on in spite of the Key Industry Duty and the advantage we have gained from the rate of exchange and ridiculously low prices are being quoted to British purchasers of bromides. The foreign bromide makers are obviously not prepared to give up this market without an intensive struggle, and the attention of the Board of Trade has been drawn to the abnormal imports of bromides in the last month or so. With proper support from British users of bromides, the British makers should be successful in their efforts to make bromides from Empire bromine. The events of the last two months have indicated that many British consumers of fine chemicals are still unaware that suitable and adequate sources of supply exist in this country, and that in most of the cases where temporary exemptions have been granted from the Key Industry Duty British makers can now see the possibility of making the products in question on a competitive basis, and are taking steps to do so.

Many people hoped that with our departure from the gold standard and the return of a strong National Government our industrial troubles were to be at an end, and that British manufacturers could count on a period of increasing prosperity. While never pessimistic, the writer feels that a complacent attitude that things will come right by themselves if we get a general system of tariffs is likely to lead to bitter disappointment. This country has a very hard time indeed ahead of it in order to secure a favourable trade balance. It is necessary that everyone should realise that only by the most energetic action on the part of industry to improve its competitive position in the world's markets will prosperity come, and that unless such action is taken tariffs will be a snare and a delusion. The British consumer must also co-operate in supporting the British producer, and here we look not only to the consumer in this country but to all consumers throughout the Empire.



WALLERSCOTE WORKS, NORTHWICH: GENERAL VIEW OF THE KILN INSTALLATION AT ONE OF THE SODA ASH FACTORIES OF IMPERIAL CHEMICAL INDUSTRIES.



A BATTERY OF OLIVER ROTARY VACUUM FILTERS, DEALING WITH BEET SUGAR CARBONATION JUICES. (OLIVER UNITED FILTERS, LTD.).

Developments in Chemical Engineering

By H. W. Cremer, M.Sc., F.I.C., M.I.Chem.E.

The author of this article is Director of Chemical Engineering Studies at King's College, London. He has surveyed the progress which has been made in materials of construction as well as developments in basic chemical engineering operations.

AN outstanding event of the year was the extremely well organised Exhibition arranged by the British Chemical Plant Manufacturers' Association in conjunction with the Chemical Engineering Group of the Society of Chemical Industry, which was held on the occasion of the Jubilee celebrations of the Society in July. The Association concentrated on the trade side with the object of showing the various constructional materials, types of plant, apparatus and general equipment available in this country for the chemical industry and the numerous branches of activity associated with it. The group, on the other hand, emphasised the research side and, with the co-operation of the Department of Scientific and Industrial Research, arranged a most valuable group of exhibits designed to show the application of various raw materials to the chemical industry, with special reference to chemical engineering. These exhibits clearly demonstrated the important part which science is playing in the development of industry, and the detailed and very careful technical research work which is essential to all modern progress. Under the depressed conditions of manufacture and trade generally, the past year might have been regarded by many as one for consolidation and even retrenchment, rather than one for progressive development. Yet those who visited the Exhibition were no doubt struck by the range of materials and plant displayed, and must have come away satisfied that British chemical plant and equipment are now obtainable to meet the most exacting requirements as regards engineering design, quality of raw materials, and thoroughness of workmanship. The whole Exhibition provided evidence of the improved technique in the design and construction of plant, and testified to the ability of its organisers and to the progressive instincts of the exhibitors.

Materials of Construction

The rôle of metals and their alloys in chemical engineering is of such general interest that the progress which has been made in increasing their utility is particularly welcome. Especially is this the case with corrosion and heat resisting materials, among which are the important nickel-chromium steels. At one time it was thought that all conditions could be satisfied by one small range in composition, but difficulties arose when this class of steel was used for many diverse purposes. As a result of the researches undertaken the austenitic nickel-chromium steels have been evolved, which steels, on account of their wide range of resistance to chemical attack, have been adopted for many varied types of chemical plant. This resistance can be increased by the addition of small amounts of other elements such as silicon and molybdenum, and tungsten where high temperatures and pressures are involved. These steels show good mechanical properties, are fairly easily worked, and can be readily welded either electrically or by the oxy-acetylene process. In connection with the welding of the material, it should be noted that the former difficulties due to brittleness and intercrystalline corrosion which were sometimes experienced after this operation, and generally termed "weld decay," have been overcome. In addition to the well known silicon-irons, cast iron may be made corrosion resisting under certain conditions by the addition of varied amounts of nickel, chromium and copper; one particular type having the nickel-copper addition made in the form of Monel metal.

Non-Ferrous Metals

Turning now to the non-ferrous materials, mention may first be made of the use of aluminium additions to brass. At one time there was considerable objection to the inclusion of aluminium in copper-zinc alloys owing to the difficulty of casting. With the improved methods of casting now in use a material highly resistant to certain forms of corrosion has been produced containing about 75 per cent. copper, 2 per cent. aluminium, and the remainder zinc. In the straight aluminium copper alloys, there is the very useful 10 per cent. aluminium bronze, which is being used for numerous purposes

in the form of die-castings, for example, slats for conveyor chains which come into contact with saline solutions. Mention may also be made at this point of a newly developed natural alloy called "Batterium," of composition copper 80 per cent., aluminium 9 per cent., nickel 2 per cent. It is claimed that this alloy is more resistant to corrosion in certain specified cases than Monel metal and various "stainless steels." The aluminium-silicon and aluminium-magnesium alloys continue to be improved in their production and working and are being more widely used. Lead containing small percentages of antimony has been used for many years for certain parts of chemical plant, but any further increase in the antimony content has increased the hardness of the lead. A lead-antimony alloy containing a high percentage of antimony is now made, however, which can be rolled into fairly thin sheets and used for lining vessels. A considerable amount of work has been carried out on the production and applications of beryllium, and progress in this field will be awaited with interest.

Use of Precious Metals

There have been interesting developments in the uses of the precious metals. The very low prices of silver which were ruling during the first nine months of 1931 brought about a growing use of this metal for commercial purposes. This was practically entirely based on the metal's powers of withstanding attack by organic acids and found its spheres of application in the industries where these acids are made or where they are handled in larger or smaller quantities in commodities such as foodstuffs. In some cases apparatus of considerable size and weighing up to a quarter of a ton has been made out of the pure metal, but more usually recourse has been had to silver lined vessels. The most striking point about the older uses of platinum is the way in which this metal holds its own in the sulphuric acid industry against the very much cheaper modern substitutes. It appears that its superior efficiency more than counterbalances the disadvantage in first cost. The question of poisoning seems to be receding into the background, the modern demand for a pure final product assuring a complete removal of all potential poisons from the gases in any case. A new use for platinum which is too recent for its future to be foreseen is the development of a commercial plating bath. The sponsors claim many advantages for platinum plate as a non-corrodible and non-tarnishable coating of pleasant appearance for articles of silver or base metal for both decorative and industrial purposes.

Processes for the protection of iron by other metallic films, capable of withstanding corrosion, abrasion, etc., have made considerable strides. In recent years the electro-deposition of cadmium and chromium have replaced to some extent the use of nickel, and now tungsten plating is a commercial proposition. It is stated that a metal electroplated with tungsten is not affected by any mineral acid and, although it is attacked by a mixture of nitric and hydrochloric acids, it is resistant to either separately. Hot dipping and metal spraying processes have also been improved, and metal coatings generally can now be applied so as to form a thoroughly reliable protection for the base metal. Important progress is to be recorded in the production of enamelled iron, particularly in the deposition of acid-resisting enamel on mild steel.

Rubber as a Constructional Material

There are notable advances to be recorded in the application of rubber as a constructional material. Para rubber sheet and ebonite have long been used, but recent researches have been directed towards the production of special rubber compounds which could be applied *in situ* by unskilled labour. Methods and materials are now available as a result of which the lining of plant on the site has become an ordinary piece of chemical engineering practice. Defects due to faulty adhesion are being overcome by special organic resinous self-setting compounds, and quicker methods of curing

are being developed. Recently, the spraying of rubber and rubber mixtures has been tried with promising results. Amongst other recent introductions may be noted acid resisting insulation and flooring with cork and rubber emulsion, the production of three-ply wood and metal coated plywood utilising rubber glues which are completely water and acid proof, and the production of rubber concrete and concrete mixtures for tank construction. The latter material is a form of artificial wood and is flexible, almost acid resisting, and can be cast in any shape. It does not expand or contract and is therefore claimed to be far superior to wood for vats which are to be rubber covered for hot acid solutions. Certain classes of rubber can be moulded with screw threads which are stout enough to make sound joints. In addition, rubber valves, rubber lined pipes and fittings, meters and pumps are all available, and investigations are in progress designed to produce plastic rubber compounds which set on exposure to air to form a rubber-like continuous sheet.

Fused Silica

Recent advances in the manufacture of fused silica articles have appreciably increased the usefulness of this material, and equipment is now available of considerably larger dimensions than formerly. Carborundum is now being used in the form of heat regenerator tubes, combustion chambers, boiler furnace walls, etc., on account of its high heat conductivity and resistance to high temperatures. Progress has also been made in the manufacture of chemical stoneware during the last few years and, although this material has largely been superseded by special metals, rubber, etc., ceramic products still enjoy a large field of application. Among the newer materials, "steatite" (soapstone) is being increasingly employed on account of its mechanical strength and comparative resistance to acids and bases before firing. It is possible to machine steatite, and to bore with an accuracy of a hundredth of a millimetre. A limitation to the use of this material is determined by its relatively high expansion coefficient. Reference may also be made to the increased use of glass silk for purposes of heat insulation. The means employed for the manufacture in this country embodies the most modern method of heating glass, and spinning glass fibres by an electrical process. A feature of glass silk, which comes from the spinning department in a large hank containing many large fibres, is its ability to divide itself when spread out into a veil like sheet. Successive sheets are spread out, one on top of the other, until the desired thickness of material is produced. The superimposed fibres form themselves into a firm mattress, which can be cut into strips or otherwise shaped to the desired form. The wrapping can be finished off with cement or canvas covering, or in other ways.

Fuel Technology

Some definite progress has been made in various aspects of fuel technology. In pulverised fuel a novel principle has been developed at the Fuel Research Station in which the coal dust is burned in a vortex so that the particles are maintained at a definite radius by centrifugal force, whilst the air passes through the dust on its way to the outlet. By this system high heat releases in a given combustion volume have been developed. It is reported that the pulverised fuel internal combustion engine which is being developed by Pawlikowski is making distinct progress.

On the hydrogenation of coal great advances are reported by Imperial Chemical Industries, and it is known that they have erected a large experimental plant at Billingham. With its ancillary processes it forms the chief technical advance in this branch of coal technology, since the process necessitates the treatment of coal with hydrogen at temperatures of about 450° C. and at pressures of from 200-250 atmospheres.

Some attention is being paid to the development of mills suitable for the treatment of coal by shatter as a preliminary to separation into the petrographic ingredients. These ingredients possess definite properties which vary with the coals treated. The separation of coal into the ingredients enables portions to be used for specific purposes.

Heating Systems

The increasing demand for reactions and processes which need to be carried out at temperatures in the neighbourhood of 300° C. has been met by the use of heating systems using hot oil circulation. In cases where higher temperatures are

required than can be obtained by oil circulation, other media for fluid heat transmission have been tried out under pressure and have been applied successfully on the large scale. Electricity is being increasingly used for heating purposes where high temperatures are required, and as the source of heat in fluid heat transmission systems.

In connection with heat exchange and condensing plant, it is satisfactory to note that increasing attention is being paid to the fundamental laws of heat transfer and fluid flow, and the research work which has accumulated and which is still in progress on this vital subject will, it is hoped, shortly provide a more solid foundation for the scientific design of such plants.

Drying Plant

Several novel types of dryer have been described, and existing types modified. Considerable progress has been made in this country in continuously operated spray drying plants, particularly for special kinds of food products. Recently the ordinary dried milk powder and foods containing this as a basis have been developed to a large extent with the incorporation of other ingredients, in particular, a very high percentage of added fat. Furthermore, increased attention is being paid to vitamin content, and the combination of these factors naturally requires an improvement in existing drying processes. It has been shown that the latest type of spray drying plant will produce very satisfactory products of this nature, whereas certain difficulties such as solubility, properties of the fats, etc., were often encountered with the older methods of roller drying and the use of spray nozzles. In many cases the original food preparation is in the form of a liquid containing a relatively small percentage of solids, and for economy in first and working costs, this is concentrated in a steam heated evaporator before being fed to the spray drier. Advantages are claimed in this connection for the continuous film evaporator, the advances made in the latter being mainly in the direction of decreasing the maximum temperature to which the liquid is exposed during its short time of contact with the heated surfaces. Further evidence of the utility of silica gel as a drying acid purifying agent are to be recorded.

Evaporation and Distillation

The use of high velocities in evaporators has again come under consideration, no doubt as a result of recent work published in America on this subject. Whilst forced circulation may possess distinct advantages in certain special cases, such as the concentration of liquids of very high density, in general it is doubtful whether the power requirements make it a commercial proposition, and it is advisable to consider each case on its own merits. In the case of food products where facilities for cleansing and sterilisation are necessary, accessibility is vital, and easy access to all parts coming into contact with the liquor is essential. Much consideration is given to such requirements in modern evaporation plant, an excellent example of which was afforded by a new forced circulation type of vacuum evaporator with external calandria shown at the Exhibition. Progress is also to be recorded in concentration by submerged-flame evaporators, in which direct combustion of the fuel takes place actually in the liquor which is being concentrated. The outstanding advantage claimed for this method of evaporation is that the heat is not transmitted to the liquor through a metal wall, and consequently the difficulties of hard-scale formation from scaling liquors are eliminated. In addition, where corrosive liquors are to be concentrated, the vessel can be lined with a corrosion-resisting material, which is not subjected to a higher temperature than the boiling point of the liquor.

Recent developments in fractional distillation have been largely in the direction of the single-flash principle. Modern tube stills have two banks of tubes, in the first of which the liquid is heated by conduction and convection from the hot gases, and in the second by radiation from furnace walls. Heat interchange efficiency is obtained by maintaining turbulent flow in the tubes. Pressure is sufficiently high to produce vaporisation in the flash chamber without further application of heat. Modern fractionating equipment consists of large bubble tray towers, from which a number of side streams are taken to re-boiling, or stripping, sections, where distillation is effected by means of superheated steam. Numerous patents have been published, dealing with the

design of dephlegmators, bubble caps, etc., the object being to promote more efficient contact between the liquid and vapour phases.

Centrifugal Separation and Filtration

Modern tendencies in centrifugal separator design are in the direction of self-discharging machines. These are now available of extremely robust construction and in very compact forms. There are great possibilities for such machines in the chemical industry, as they are adaptable for treating a larger variety of products at a very rapid rate.

A novel vertical pressure leaf type of filter was shown at the Exhibition. In this design, the whole battery of leaves may be drawn forward, thus permitting the cake to be scraped off or shaken directly into a truck placed beneath the filter. The covering door is deep from back to front to allow space for two cantilever supports carrying the leaves, the door being secured by a toggle or screw closing gear. Freedom from leakage is ensured, the filter having only a single external joint. The use of the principle of edge filtration has also developed during the year. It is claimed that owing to their simplicity of operation, and compactness, filters of this type are convenient for use in directions where previously filtration has been especially difficult. Progress is also noticeable in continuous rotary vacuum filtration, where very large volumes of liquor have to be handled. Staybrite wire can be drawn in the form of very fine wire and made up into the form of gauze, and Monel metal wire cloth is also available as a filter medium. Developments are also recorded in the use of ebonite as a filtration material. A preliminary vulcanisation is not carried past the point at which the material can be powdered in a grinding mill. After grinding, the powder is put into pre-warmed iron moulds and the vulcanisation completed under pressure. In this way very porous materials are obtained, and investigations are in progress to ascertain whether still finer products, for use in bacterial and colloidal filtration, can be obtained.

Several patents have been granted covering the separation and classification of materials by elutriation. These classifiers operate on well-known principles, and differ only in details such as methods of discharge.

Size Reduction

In countries producing cement from hard materials in the wet way, interest has been aroused by the adoption of closed circuit grinding of the raw materials, with resulting economy in power costs. Several proposals have been made in connection with the use of combined disintegrating and screening plant by fixing rubber-tyred wheels or rollers above a screen. The process is applicable to materials which can be disintegrated by tearing or rubbing. High speed colloid mills may be divided into two classes, viz.: (1) beater mills, and (2) mills in which the material is forced through a slit as a thin film by high speed rotors. Recent improvements in the first class are mainly concerned with increase of speed and reduction of power requirements. In the "vacuum dryer colloid mill" improved results are obtained by forcing the liquid in counter-current to the direction of movement of the beaters.

The process of mechanical atomisation of liquids by means of rotating discs, etc., has received considerable attention, and applications of this principle are to be found in spray dryers, gas treatment plant, evaporators and sulphuric acid plants.

High Pressure Processes

The accomplishment of high pressures in the nitrogen industry has led to the establishment of a new technique which has had far-reaching effects in other industries. The construction of plants for the catalytic hydrogenation of heavy petroleum oils, working at pressures of between 250 and 300 atmospheres and at temperatures of over 480° C., is an accomplished fact, and cracking processes are now in operation at considerable temperatures and pressures. As already stated, recent developments have justified the confidence placed in the coal hydrogenation process, and many other industries have benefited as a result of the advances in high pressure and high temperature technology. Reference must be made here to the extremely valuable work carried out in this field at the National Physical Laboratory, some interesting results of which were shown at the Plant Exhibi-

tion in July. Mention should also be made of the Hunter Memorial Lecture on "Organic Synthesis Facilitated by Pressure" which was delivered by Professor G. T. Morgan earlier in the year. It is very satisfactory to note that manufacturers in this country are in a position to provide the necessary heat exchangers and vessels for reactions at high temperatures and pressures of excellent materials and form, and can challenge any other country in the world in this respect.

Recording and Control Equipment

Far more attention has been given in recent years to exact control of chemical and mechanical processes. Among the more important factors requiring control are temperature, pressure, humidity, composition as applied to the gas phase and to hydrogen ion concentration, weight and volume, flow, specific gravity, liquid level and time. Such factors are now generally capable of control by automatic means, notably in the form of distant reading devices mustered in control panels, and there is increasing evidence to show that industry is taking advantage of the recent progress in this direction. In this way really accurate data are being obtained which are not only of great use in the control of plant, but also in the investigation of the actual process itself. There can be no doubt that chemical engineering will, in general, benefit greatly by the extensive and reliable data which can be obtained by this means.

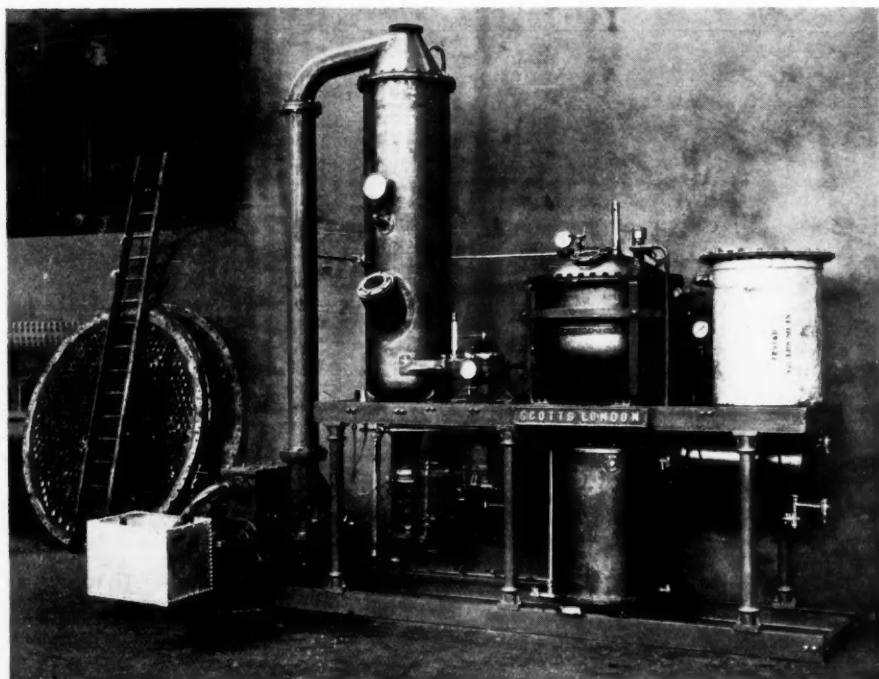
Chemical Engineering Education

No review of this kind would be complete without reference to the important developments which have taken place in providing facilities for the organised teaching of chemical engineering in this country. Full tributes have already been paid to the late Professor J. W. Hinchley, whose pioneer work in and intense devotion to this subject at Imperial College, London, and elsewhere are fresh in the minds of all. The Institution of Chemical Engineers, of which the late Professor was one of the founders and which he served so faithfully and so well, is paying particular attention to the educational side of its activities. Evidence of this fact was afforded during the year at the ninth annual corporate meeting of the Institution, when, in place of the usual presidential address, Mr. J. Arthur Reavell initiated a discussion on the training of a chemical engineer. Although much doubt still appears to exist as to whether chemistry or engineering should form the chief foundation on which a chemical engineering education should be built, yet most people have now come to agree upon the need for the product of such education in modern industry. Whether or not it will be possible to create an undergraduate training in this subject, which will be acceptable to all those who hold such conflicting views at present, remains to be seen. Meanwhile, definite progress is being made as regards post-graduate courses already in existence. It is particularly gratifying to note the large developments at University College, London, where Prince George recently performed the opening ceremony of the new building for the Ramsay Memorial Laboratory of Chemical Engineering. It is likely that a considerable extension of existing facilities will soon be possible at King's College also, where very valuable experience of the teaching of the subject has been gained during the past four years.

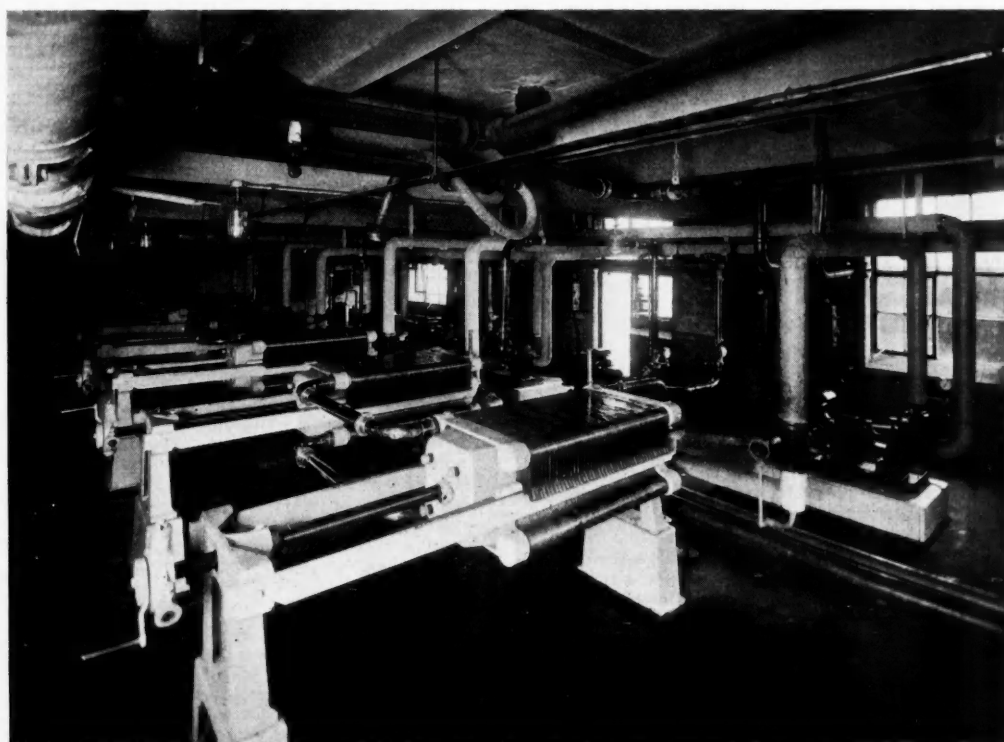
Indian Institute of Science

Growing Importance in Service to Industry

THE work of the Indian Institute of Science at Bangalore is growing in importance and value year after year. The settled policy of the Institute has been to render all possible assistance in solving problems which have some bearing on industry. But it is not practicable in every case to adapt these to an educational purpose, and the requirements of students desiring instruction in methods of research are recognised as the first consideration. The total number of students undergoing training in research during the year 1930-31 was 133, coming from all parts of India. Considerable additions have also been made to the equipment in all departments, which now provide opportunities for advanced instruction, which are not only unrivalled in India but compare favourably with the best institutions overseas. The Institute library now contains 19,000 volumes describing modern research, and French and German classes are regularly held to enable students to use the library fully.



THE SCOTT PATENT FORCED-CIRCULATION EVAPORATOR FOR DELICATE EXTRACTS.
(GEORGE SCOTT AND SON, LONDON, LTD.).



A BATTERY OF "A.P.V." PATENT (PLATE TYPE) HEAT EXCHANGERS.
(ALUMINIUM PLANT AND VESSEL CO., LTD.).

The Nitrogen Industry in 1931

By E. B. Maxted, D.Sc., Ph.D., F.I.C.

Papers and patents relating to progress in the Nitrogen Industry during the past year are reviewed in this article. The chief features dealt with are the synthesis and oxidation of Ammonia, and manufacture of Ammonium Salts.

THE present year has, as in most other industries, been a period of low prices, and a time—having regard to the decreased demand due to the world-wide depression of trade—of over-production. Thus, the current price of ammonium sulphate, namely about £6 15s. per ton, is of an order between one third and one quarter the peak prices of the years immediately following the war; and, while few commodities have fallen so greatly in relative selling price, the possibility of producing apparently profitably at this figure is a striking tribute to the economic and technical advances which have been made in the synthesis within the past decade, combined possibly with the passing of the initial period of specially high effective incidence of capital charges in the older factories.

In view of considerations based on the steady increase in demand during more normal years, even at higher prices, it would appear probable that the lack of buying is due rather to world poverty than to real saturation of the normal agricultural needs; indeed, the whole fabric of the inordinate growth of the industry has been based on what would seem to be a sound economic view of the increased earning power possible by even greater use of nitrogenous fertilisers.

For the above reasons, there has been some restriction of output and relatively few extensions to plant or new factories; although it is interesting to note that the erection is reported of two Fauser plants, one for the fixation of 100 tons of nitrogen per day in the Donetz Basin and the other by the Consolidated Smelting and Mining Co., in Canada.¹ Of other movements tending to control output and prices and to localise sales, reference may be made to the restrictions on imports of nitrogenous fertilisers into many countries² and to the efforts which have been made towards an international nitrogen agreement.³

Synthesis of Ammonia

A very readable outline summary of the development of the nitrogen industry has been given by G. C. Riley and H. G. Bott.⁴ This includes a good general description of the Mont-Cenis, Casale and Billingham plants, and is recommended as a most interesting general survey. For a more detailed account of the Mont-Cenis process, which is characterised by the relatively low temperature and pressure at which the synthesis is carried out, reference may be made to an article by W. F. Scholvien.⁵ The capital cost of the complete plant is stated to be, under present conditions, less than 50 dollars per ton-year of nitrogen for a plant of moderate size. In this process, the hydrogen required is separated from coke-oven gas by low-temperature fractionation in a Linde-Bronn plant. It may be noted that the Mont-Cenis factories near Dortmund have been leased to the German Interessen-Gemeinschaft.⁶

Several patents dealing with the purification of the gases have been published. R. S. Richardson⁷ recommends scrubbing the gas mixture used for the synthesis with liquid ammonia. A somewhat similar process⁸ consists in adding a small percentage of ammonia to the gas before compression. On compression this ammonia liquefies and entrains many of the catalytically inhibitive impurities present. In another method, which is used for the purification of gases containing ammonia, carbon dioxide is added in amounts not exceeding 50 per cent. of the ammonia present.⁹ Ammonium carbonate is formed, which, when condensed, removes moisture and suspended matter before the re-passage of the nitrogen-hydrogen

mixture over the catalyst. To prevent the blockage of pipes by ammonium carbonate from circuit gases containing carbon dioxide, the make-up gas may be mixed with ammonia and moisture in quantities sufficient to dissolve the ammonium carbonate present.¹ Attention may also be drawn to a variation in the preparation of a nitrogen-hydrogen mixture by the continuous catalytic method involving the interaction of carbon monoxide and steam. In a patent granted to D. Tyrer and Imperial Chemical Industries, Ltd.,² producer gas, without the usual condensation of the excess of steam which is always present, is passed over a catalyst for the above conversion. The process would seem to bring with it a valuable economy in the steam required for the catalytic replacement of carbon monoxide by hydrogen.

Less work than usual appears to have been done on new catalysts for the synthesis of ammonia; although A. T. Larson³ recommends a contact body consisting of an iron base, promoted with compounds of potassium and magnesium and containing oxides of chromium, titanium, vanadium, etc. Finally, some details of ammonia compressors have been published by Schneider⁴; and safety precautions in high pressure plants generally, particularly during repairs and adjustments have been discussed by R. J. Woods in the course of the Chemical Session of the Safety First Congress.⁵

Oxidation of Ammonia

Webb⁶ has discussed the factors controlling the yield of nitric acid in the above reaction. In particular, the results obtained with multiple gauze catalysts were examined, and it was found that catalysts of this type presented no material advantage over those composed of a single gauze. The production of nitrogen and consequent loss of yield is considered to be due principally to the interaction of nitric oxide with ammonia and only to a small extent to the decomposition of nitric oxide.

Several papers dealing with the use of increased pressures have appeared during the year. Thus, a description of low and high pressure plants for the oxidation of ammonia, as used by the Du Pont Company, has been given by G. B. Taylor, T. H. Chilton and S. L. Handforth.⁷ Platinum gauze catalysts are used; but the losses from platinum-rhodium gauzes are stated to be less than from platinum. A high pressure plant involves a lesser initial cost but a higher power consumption than is required for a low-pressure system. An increased pressure which, in the plant described, was of the order of eight atmospheres, facilitates however the production of concentrated acid. The use of pressure has also been dealt with by Fauser.⁸ In this plant, in which turbo-compressors of chromium steel are employed, no mechanical troubles in compressing were experienced; but it is stated that the efficiency and the life of the catalyst are reduced by pressure, although it helped the condensation side of the process. If pressure is used, its most effective value is about 5 atmospheres, in conjunction with a gauze temperature of 850°-950° C. In a further patent,⁹ Fauser oxidises ammonia at ordinary pressure, cools the reaction mixture by passage into a waste heat boiler of large surface and small volume, and only after this stage compresses it in turbo-compressors, as above. The mixture is further cooled, with deposition of water, following which the nitrogen peroxide is absorbed in the usual way. The condensation of nitrogen oxides under pressure is also dealt with in two patents of

¹ The Chemical Age, **25**, 219.

² Chemistry & Industry 1931, 728.

³ The Chemical Age, **25**, 149.

⁴ The Chemical Age, **24**, 161 and 188.

⁵ Chem. & Met. Eng., 1931, 38, 82; The Chemical Age, **24**, 304.

⁶ The Chemical Age, **24**. Dyestuffs Monthly Supplement, p. 3.

⁷ U. S. Pat. 1,765,541.

⁸ U. S. Pat. 1,765,534.

⁹ Jones, U. S. Pat. 1,794,903.

¹ Societe l'Air Liquide.

² Brit. Pat. 347,601.

³ U.S. Pat. 1,771,130.

⁴ Brenstoff Chemie, 1931, **12**, 273; ex-Brit. Chem. Abs., B., 1931, p. 923.

⁵ The Chemical Age, **24**, 513.

⁶ J. Soc. Chem. Ind., 1931, **50**, 128T.

⁷ J. Ind. & Eng. Chem., 1931, **23**, 860.

⁸ Chim. et Ind., 1931, **25**, 556.

⁹ Brit. Pat. 345,662; The Chemical Age, **25**, 10.

Caro and Frank.¹ In the latter of these, liquid nitrogen peroxide is condensed out at a temperature of 70°-120° C. and at a pressure between 50 and 200 atmospheres. Subsequent rectification of the liquid nitrogen oxides and their conversion into nitric acid may be carried out at the ordinary pressure. A further process, also due to Caro,² is of the type in which water is preferentially condensed out of the reaction mixture with as little as possible oxidation of the nitric oxide. The oxides of nitrogen subsequently formed are then liquefied.

While platinum seems to be firmly established as a catalyst in the reaction, the tendency of vanadium to replace this metal in other industries is apparently indicated by a patent of the Selden Company, in which the use of zeolites as supports for catalytically active materials is suggested. Zeolites containing vanadium have already found employment in the sulphuric acid industry; and, although these zeolite supports may obviously be used for a large variety of catalytically active bodies, they would appear to be particularly suitable for vanadium in its application to ammonia oxidation. Of other forms of catalyst, mention may be made of the use of hammered or rolled platinum, for instance in gauze form, in place of ordinary platinum³ and to a suggestion of Smith and Imperial Chemical Industries, Ltd., according to which the catalyst gauze employed is supported by means of an arrangement of silica rods.⁴

Finally, attention may be drawn to an interesting proposal by Ruhr Chemie A.-G. for the direct manufacture of ammonium sulphate-nitrate⁵ by the combined oxidation of ammonia and sulphur dioxide. The ammonia is first oxidised in the ordinary way, after which the reaction gas is mixed with sulphur dioxide and any necessary steam. Ammonia and the mixture resulting from the above oxidation are passed in the proper proportions into a saturated solution of ammonium sulphate and nitrate when solid ammonium nitrate-sulphate crystallises out.

Ammonium Salts

Several patents dealing with the manufacture of ammonium sulphate by the oxidation of sulphur dioxide by means of air in the presence of ammonia have been published. Thus, D. Tyrer and Imperial Chemical Industries, Ltd.,⁶ allow sulphur dioxide and ammonia to interact in the presence of a limited amount of water, together with a small percentage of nitrogen oxide, when solid ammonium sulphate is deposited. A somewhat similar process is described by the Bataafsche Petroleum Maatschappij.⁷ In a further patent,⁸ also of the latter company, dry sulphur dioxide and ammonia, in the proportion of one to two by volume, are passed into tubes heated internally to 100°-120° C. and cooled externally, when the compound $\text{NH}_4\text{SO}_4\text{NH}_2$ condenses out and is removed as a fine powder. This is subsequently converted with moist air into ammonium sulphate, a suitable catalyst for this last stage being iron sulphate. Mention may also be made of a process, due to G. Fauser⁹ for the manufacture of dry ammonium sulphate by utilising the heat of neutralisation. According to this method, gaseous ammonia and 65 per cent. sulphuric acid are sprayed in the calculated proportions at the top of a tower. The heat of reaction causes the evaporation of the water; and dry ammonium sulphate is deposited and may be conveyed away directly.

The technology and economics of ammonium sulphate manufacture from calcium sulphate in place of from free sulphuric acid have been re-discussed in a particularly clear manner by Parrish.¹ The article includes tables showing the relative costs of the two methods under various conditions. There would appear usually to be a definite economic advantage in employing the calcium sulphate method, unless abnormal transport factors are involved; and, under proper conditions, the double decomposition involved is stated to present

no practical difficulties. For details, reference should be made to the original article.

Miscellaneous Nitrogen Compounds

Within the past few years considerable work has been done on the manufacture of hydrocyanic acid by the interaction of ammonia and hydrocarbon gases at high temperatures. This has been dealt with by H. Küster² with special reference to the present state of knowledge of the reaction. It may also be noted that the deposition of carbon usually observed in the process may be eliminated by diluting the gases with hydrogen.³

The manufacture of cyanamides is treated in a patent of N. Caro and A. R. Frank.⁴ Calcium or magnesium carbonate is allowed to react with ammonia, the corresponding cyanamide being produced. The water formed is removed by suitable absorbents, for instance by phosphorus pentoxide, mixed with the briquettes treated. If this particular absorbent is used, a fertiliser containing cyanamide and phosphoric acid is obtained. A further patent involving the treatment of calcium carbonate with ammonia and carbon dioxide at 600°-800° C., due also to Caro and Frank,⁵ is stated to lead to calcium cyanamide of high percentage purity. The production of urea from carbon dioxide and ammonia is represented by a patent of the Interessen-Gemeinschaft,⁶ which deals with a method of checking the decomposition of the product. This is effected by distillation in the presence of an excess of ammonia.

Finally, two patents leading to the production of fertilisers may be mentioned. The Interessen-Gemeinschaft⁷ have protected a special fertiliser made by melting together urea and calcium nitrate in the molecular ratio of 4 to 1, the melt being sprayed in order to obtain the mixture in a granular form, and the preparation of the potassium nitrate itself by the heating together of potassium chloride and aluminium nitrate has also been described.⁸ In the latter case, the heating is continued until no more oxides of nitrogen or chlorine are evolved, when the mass is lixiviated with a solution containing potassium nitrate.

If the above work be compared with that carried out during a typical year of, say, a decade ago, it will be seen that the synthesis of ammonia has now become more or less stabilised, in that suggestions for modifications in the catalyst or in the construction of the plant now appear far more seldom. Similarly, the large number of papers and patents dealing with the production of neutral ammonium sulphate has disappeared, indicating, in this case also, the attainment of satisfactory works practice. In the synthetic ammonia industry the principal economic problems to-day lie probably in the first place, in the manufacture of the gases—namely, in the choice between high-temperature chemical methods and low-temperature separation methods in the treatment of a suitable fuel gas—and, secondly, in the conversion of the ammonia to the most suitable salt. In this connection, the various proposals published this year for the direct production of solid ammonium sulphate will be noted with interest.

Indian Chemical Trade in 1930-31

THE Government of India Review of the trade of India for the year 1930-31, which is now available, puts the total imports of chemicals into India (excluding manures and medicines) at Rs. 261 lakhs in value, as against Rs. 279 lakhs in the previous year. Soda compounds accounted for 45 per cent. of the trade. The United Kingdom was the chief supplier. Caustic soda showed considerable increase. Imports of acids were practically stationary during the year at 35,000 cwts. The supplies of nitric acid doubled in quantity but sulphuric acid declined. Ammonia and ammonium compounds declined to some extent but the supplies of bleaching materials registered a very satisfactory increase. Taken as a whole the shares of the United Kingdom, Italy, Norway and Japan declined, while Germany and the United States considerably improved their position.

¹ Brit. Pats. 338,566 and 342,068; The Chemical Age, **24**, 78 and 421.

² Brit. Pat. 337,847; The Chemical Age, **24**, 34.

³ Lost, Brit. Pat. 346,247; The Chemical Age, **25**, 40.

⁴ Brit. Pat. 346,800; The Chemical Age, **25**, 75.

⁵ Brit. Pat. 349,998; The Chemical Age, **25**, p. 279.

⁶ Brit. Pat. 342,524; The Chemical Age, **24**, p. 446.

⁷ Brit. Pat. 341,570.

⁸ Brit. Pat. 346,508; The Chemical Age, **25**, p. 74.

⁹ Chem. & Met. Eng., 1931, **33**, 546.

¹ The Chemical Age, **24**, 23.

² Brennstoff Chemie, 1931, **24**, 329.

³ Imperial Chemical Industries and others, Brit. Pat. 353,407; The Chemical Age, **25**, 431.

⁴ Brit. Pat. 338,023; The Chemical Age, **24**, 56.

⁵ Brit. Pat. 341,299; The Chemical Age, **24**, 354.

⁶ Brit. Pat. 337,394; The Chemical Age, **24**, 12.

⁷ Brit. Pat. 338,644; The Chemical Age, **24**, 78.

⁸ Kaliforschungsanstalt, Brit. Pat. 343,796.

Developments in the Chemical Industry During 1931

By Colonel G. P. Pollitt

Colonel Pollitt is a director of Imperial Chemical Industries, Ltd. The activities of I.C.I., extending to a very wide range of products, which are destined for use in all branches of industry, enable him to give a concise summary of the notable developments which have taken place.

ANY review of the trade of 1931 must naturally revolve round the abandonment by this country of the Gold Standard, which was the most important single economic event of the year. This abandonment was only effected three months ago but in the short time that has elapsed British industry has undoubtedly received a stimulus which has been especially noticeable in the chemical trade where monthly figures for sales have definitely shown an upward tendency in most branches. Naturally the chemical industry stands in a specially favourable position. It benefits by the decreased cost of exports to the foreign purchaser, while it does not suffer from the necessity of increasing production costs because it depends very largely on raw materials produced within the British Isles.

Two specific examples of the way in which the chemical industry has benefited by the currency position may be found in the sale of alkali products in Japan and of saltcake in Scandinavian markets for the sulphate pulp industry. Although that industry has suffered from labour disputes during the year, the depreciation of Scandinavian currencies, in terms of the dollar, will enable the Scandinavian sulphate pulp industry to compete more successfully in world markets than their American rivals. This benefit to the British saltcake industry at the present is enhanced by the remainder of the European suppliers, particularly Germany, having a dearer currency. It must, of course, be remembered that the saltcake market is the subject of international agreement.

Obviously there are grave disadvantages in a depreciated currency. A devaluated and unstable pound may ultimately have the effect of further paralysing world trade, so that, although we may enjoy a slightly increased share of world export trade, the actual volume and value of our trade is diminished. On the other hand it is, of course, possible that rising prices will stimulate trade and that the British exporter's temporary advantage will prove an initial fillip to a more permanent improvement.

The Abnormal Importations Act

It is too early to assess the effects on the chemical industry of the Abnormal Importations Act. Fine chemicals are already protected by the Key Industries Duties and dyestuffs by the Dyestuffs Import Regulation Act, which, with the consent of the Colour Users' Association has been extended for a further year by inclusion in the Expiring Laws Continuation Act. All that can be said with any degree of certainty about the new Act is that in the main its effect on chemical industry must be considered as secondary and indirect. The increased manufacture in this country of products coming within the scope of the Act may, for example, lead to the increased consumption of chemicals, but there are other factors to be taken into account, and until it is known what products the Board of Trade will select in its further lists, no very useful conclusions can be drawn.

Strenuous efforts were made during the first part of the year to find a permanent basis of co-operation among the nitrogen producers of the world, but it unfortunately proved impossible to reconcile the claims of shares in the trade put forward by the various groups. Since July, therefore, there has been a large degree of competition in the world markets for nitrogenous fertilisers, accompanied naturally by a heavy fall in prices. This accelerated the tendency for gas and coke works producing ammonium sulphate to go out of production. Another aspect of the world depression has been a decrease in the demand for metallurgical coke, which has also caused a falling off in the production of by-product ammonium sulphate. The synthetic producers are therefore able with their lower production costs, to take over an increasing share of the market which has formerly been supplied by the by-product producers. They have also been better equipped than the Chileans to reduce their prices, so that recently the difference in price between synthetic nitrogen and Chile nitrate has increased. The very serious diminution in the demand for sulphuric acid as a result primarily of the

closing down of by-product ammonium sulphate manufacture, has caused the centralisation of the manufacture of the acid in the South Wales area.

Owing to the favourable prices of anhydrous ammonia, (and to the fact that for a given quantity obtained from a hydrogen cylinder, the weight of metal which has been transported and handled is about twenty times as great as when the same quantity of hydrogen is obtained by cracking anhydrous ammonia) this product has now become a cheap source of hydrogen whenever the gas is required in relatively small amounts. It is no more anomalous to spend money on the chemical process of combining hydrogen with nitrogen for the purpose of distributing it, than it is to spend money on the physical process of hermetically sealing foodstuffs in cans. In both cases the process is reversed before the product is used. The ammonia is catalytically decomposed at 600°C. in a special ammonia cracking apparatus, which is supplied by Imperial Chemical Industries, Ltd. Since the nitrogen is completely inert, the cracked ammonia may be used for most purposes in the same way as hydrogen itself. In actual practice, after allowing for depreciation, repairs, power consumption and operating costs, this source of hydrogen almost always works out more cheaply for such purposes as welding, plate-cutting, brazing and metal-spraying. By burning the cracked ammonia with the correct quantity of air, a convenient source of nitrogen is also available.

International Hydrogenation Patents

An outstanding event in the chemical industry during the year was the formation last April of the International Hydrogenation Patents Co., Ltd., representing a fusion of the interests of the Standard Oil Co., the I.G. Farbenindustrie, the Royal Dutch Shell Group and Imperial Chemical Industries, Ltd., in the hydrogenation process for the liquefaction of coal and the refining of oils in all countries of the world outside the United States and Germany. The patent rights are operated by the International Hydrogenation Engineering and Chemical Co., the staff of which is drawn from the technical staffs of the interested companies, and which is the channel through which the skill and experience of these groups will be made available to all users of the hydrogenation process.

Under the stimulus of this agreement among other factors considerable advancement has been made during the year in the production of oil from coal by the hydrogenation process. At the present time the total yield of petrol of the highest marketable quality from coal is 62 per cent. by weight, the balance being made of hydrocarbon gases, a liquid containing oxygen, nitrogen and sulphur and a small residue. The conditions can be altered so that if desired heavier oils can be produced. Including the amount required for burning in boiler furnaces, etc., the consumption of coal is 3.65 tons to produce 1 ton of petrol. If the commercial manufacture of petrol from coal is decided upon the most efficient plant would be one designed to make 200,000 tons of petrol a year at an estimated works cost of 7d. per gallon. This would provide about 8 per cent. of the total petrol consumption of the country. While world over-production and the consequent price war have resulted in the prices of imported petrol being lower than the price at which petrol can be produced profitably from coal, there is, as Mr. K. Gordon said at a recent meeting of the Oil Industries Club, ample justification for the erection of a 200,000 ton a year plant at some future date in the national interest, both economic and strategic.

Simultaneously with these developments, Imperial Chemical Industries has not been blind to the increasing importance of the Diesel engine. Considerable progress will undoubtedly be made in the future on the purely mechanical side, but improvements in the fuel itself will also be necessary. A non-toxic fuel accelerator soluble in all proportions in oil, fully protected by patents, has therefore been developed with a view to enable existing grades of oil to meet the requirements of



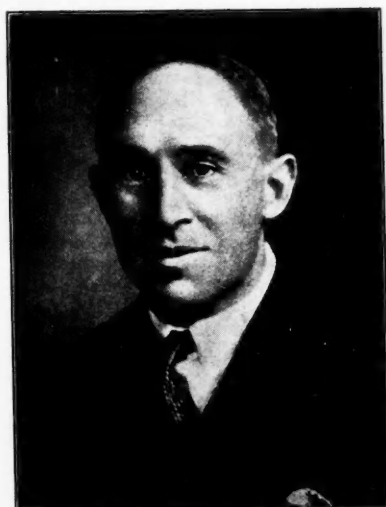
Mr. H. W. Cremer, the new Hon. Secretary of the Institution of Chemical Engineers.



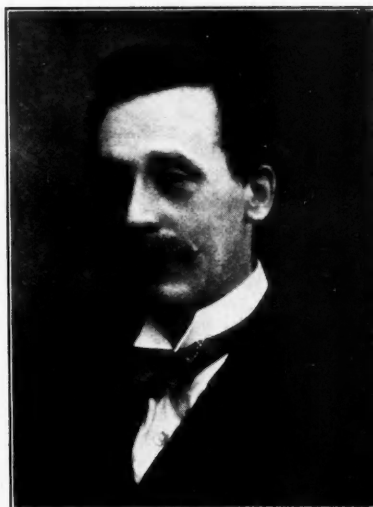
Mr. W. A. S. Calder, President, Institution of Chemical Engineers.



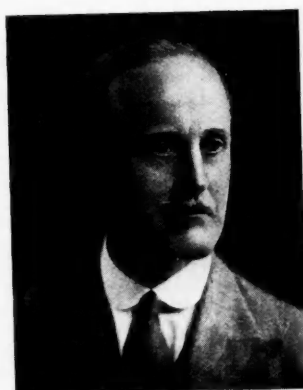
Mr. George Gray, Chairman of the Chemical Engineering Group.



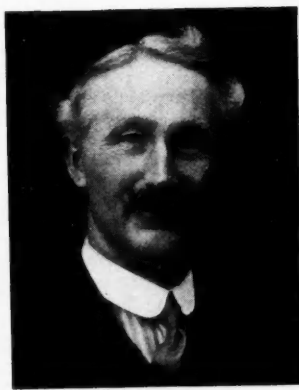
Dr. Herbert Levinstein, President of the Chemical Club.



Professor G. T. Morgan, President of the Society of Chemical Industry.



Dr. H. J. Bush, Chairman of the British Chemical Plant Manufacturers' Association.



Mr. Noel Heaton, President of the Oil and Colour Chemists' Association.



Mr. D. McDonald, the new Hon. Secretary of the Chemical Engineering Group.

the highest speed Diesels and to render possible the construction of smaller engines operating on higher speeds. The addition of the fuel accelerator reduces the spontaneous ignition temperature of the oil to which it is added, with the result that it ignites immediately on injection and combustion proceeds in a controlled manner throughout the injection period, so that the rate of rise of pressure per degree of crank angle is comparable to that in the petrol engine. The accelerator also by its explosive action increases the turbulence, and brings about a rapid mixture of air and oil, so that improved combustion is obtained with economies in fuel consumption. The same tendency to operate at higher speeds is also, of course, apparent in the ordinary petrol engine. Tetraethyl lead has solved anti-knock problems up to a certain point, but in the continual search for a higher compression ratio, this compound has not solved all problems, and at the present time the admixture of alcohols, and particularly of methanol, with petrol is of increasing importance.

The pyrolytic formation of benzol from hydrocarbon gases has passed the preliminary stage of development, and is ready for the stage of pilot plants or even of small commercial plants. The chief problem which research has satisfactorily solved was the difficulty of providing suitable materials to stand up to the higher temperatures used in the process.

Progress in Clean Coal

Progress has also been made in the preparation of clean coal, which can in certain aspects be regarded as coming within a review of the chemical industry. Various processes, such as those of the Birtley Co. and Lessing, are, already operating for both the dry and wet treatment of coal, but special reference should be made to the petrographic treatment. The recognition that coal has four distinct constituents has enabled their fractional separation to be achieved through elastic percussion crushing methods, so that it is possible to produce fines with an increased content of vitrain and clarain, and nuts and smalls with an increased durain content. Fusain can be separated economically by air suction. This separation of coal may have far-reaching effects in the future on the development of hydrogenation and low temperature distillation, as well as the coking of coal.

Solid Carbon Dioxide as a Refrigerant

Solid carbon dioxide is now being marketed in the United Kingdom under the name of Drikold. This is manufactured from by-product carbon dioxide by the Carba process in which liquid carbon dioxide is injected into a chamber near the triple point. A damp CO_2 snow is formed which freezes into a solid block. Its main field of application is, of course, refrigeration, where it has definite advantages in comparison with water ice. Much lower temperatures can be obtained, and less refrigerant is required for a given amount of cooling. This removes the necessity for re-icing during the transport of foodstuffs over long distances. Corrosion troubles, caused by water or brine, are removed and the absence of liquid makes the use of better insulating materials possible. The perfect dryness of the gas given off by Drikold tends to prevent the growth of mould. In household refrigeration Drikold may be kept in blocks and protected by insulating material. Servicing problems are relatively simple, since a block will last from 5 to 14 days.

Among the industrial applications of Drikold is its use in the ice cream trade, where it has obvious advantages over the ice-salt freezing mixtures used in the distribution of this product. It may also be used for ice cream storage. Another important application is in the transport of frozen meat and fish in insulated containers which can be transferred from railway to road trucks. Drikold is the ideal refrigerant for quick-frozen food products, and it has been suggested that a large quantity of frozen meat now sent to this country in the form of carcasses may in future be cut into joints and quick frozen in the country of origin. This would result in a saving of space and freight costs and the quality would approximate to that of home-killed meat. Other obvious applications of Drikold are in the dairy industry and in the solution of the cooling problems of brewers. The disadvantage of solid carbon dioxide which has often been mentioned, i.e. the danger of blisters from careless handling, appears to have been exaggerated. Attention is, however, drawn to this point on the packages.

In the alkali field there have not been many outstanding technical developments. A new form of sodium carbonate, of a very high degree of purity has been placed upon the market,

for the manufacture of bath salts and similar products for use in tropical climates. This will probably replace previous grades which suffered from the disadvantage of efflorescence.

The control of the sulphur content of cast iron is probably the most important problem encountered in modern foundry practice. Serious difficulty is experienced in satisfying the requirements of modern specifications because of the increased sulphur content of finished castings which result from the use of scrap metal and the introduction of sulphur from the coke in the cupola. A method which affects the desulphurisation of the metal during the process of manufacture is now available, based on the use of soda ash. By using a relatively small amount of this chemical it is possible to cheapen the cost of production of high quality castings, by enabling pig iron and coke of greater sulphur content to be used.

Chemical Exports from Russia

An, at present, relatively unimportant but at the same time disturbing factor in the chemical trade of the world is the increase of low priced Russian exports of alkali products, particularly soda ash, caustic soda, bicarbonate of soda, sodium sulphate and bleaching powder. In 1929 these exports were negligible, in 1930 most of the products appeared on the market for the first time, and in 1931 every product has shown a definite increase over 1930. There is no doubt that the reports are reliable which indicate that Russian consuming industries of these products, such as the glass industry, are being starved to enable the exports to be made. The Russian tactics are spasmodic in character and seem to be not to concentrate in any one market, but to enter all markets with small quantities of goods, and their price policy seems in the nature rather of an irritant than of definite ordinary competition.

The slump in world prices has meant that gold has become more valuable in terms of other commodities, and there has been an incentive for the gold industry to produce larger quantities of this metal. This has reacted favourably on the manufacture of sodium cyanide used in its extraction. Liquid hydrocyanic acid is now being made under carefully controlled conditions on the large scale for the first time in this country. The process used is the orthodox one, using sodium cyanide produced by the Castner process and sulphuric acid. In the hands of fumigation experts, the use of properly stabilised hydrocyanic acid is remarkably safe and there is no doubt that this country will soon follow the lead set in America in the general fumigation of ships and warehouses.

In the chlorine branch the general tendency for the displacement of bleaching power by liquid chlorine in the production of bleach liquor has continued, and interest has also been taken in the use of chlorine products in sewage treatment and fly control. Since the abandonment of the gold standard the revival in artificial silk production has stimulated the demand for liquid chlorine.

The Dyestuffs Industry.

The dyestuffs industry was further consolidated during the year by the absorption of the British Alizarine Co., Ltd., in the Dyestuffs group of Imperial Chemical Industries. Considerable increase in the range has been made by the addition of some fifty dyestuffs hitherto not manufactured in Great Britain. These additions cover every field of dyestuff and include Duratol Green G., which enables a green to be produced by the Ice-colour process for the first time. Duranol Navy Blue E and Duranol Black E for the dyeing of cellulose acetate rayon are characterised by quick absorption. A small range of Fastel colours have been placed upon the market for the manufacture of printing ink pigments of brilliant hue which are fast to light.

Coomassie brown CS and Coomassie red PGS are notable additions to the acid milling colours for wool, a series of colours has been introduced for the covering of fades in garments by garment dyers, and important additions have been made to the Chlorazol, Caledon, Thional and Monolite series of colours. Progress has also been made in the range of rubber accelerators, anti-oxidants, softeners, etc. Products of considerable assistance in the textile industry have been introduced, namely, Perminol KB for use in kier boiling. Perminol MERC as a wetting agent, particularly for caustic liquors in the mercerising of cotton cloths, and Whitcol SE for the oiling of wool.

Advances have been made in the use of chemicals in the sphere of pest control. Substances already established in

the textile trades as wetting, spreading and penetrating agents of the type prepared from sulphonated hydrocarbons and their condensation products in both the aliphatic and aromatic series, have been adapted to entomological and mycological problems. The efficiency of the already established insecticides and fungicides when used in conjunction with the former group of substances, which have a so-called activating effect, is enormously increased.

Safety Glass

The Triplex Safety Glass Co., The British Xylonite Co. and Imperial Chemical Industries Ltd. have joined forces in the search for a British-made celluloid to replace that formerly obtained from abroad, for use in the manufacture of safety glass. Their efforts have been completely successful by the development of a dense nitrocellulose, and the British celluloid which is now available is superior to the foreign product in respect of its clarity and keeping properties. The main application of nitrocellulose lacquers until recently was the finishing of the exteriors of motor-cars, but there has been a rapid development in the application of nitrocellulose finishes to leather. A nitrocellulose cement has also been evolved for sticking on the soles of light shoes and slippers much more cheaply and neatly than by the ordinary stitching methods.

A new development during the year which will have a beneficial effect upon the consumption of chemicals is the establishment in this country for the first time by the British Bemberg Co. at Doncaster, of a plant to produce about 9,500 lb. of cuprammonium yarn per day. Rapid developments are taking place in the Dulux paints and varnishes which are of a glyptal basis, and can be applied in cases where nitrocellulose is not suitable.

Explosives

The range of low freezing blasting explosives containing ethylene glycol dinitrate in the nitroglycerine has been extended by the introduction of more powerful compositions which will propagate a detonator wave more easily than the older permitted explosives. The shattering effect of high explosives is far greater than that of black powder or

Bobbinite. This fact is of importance in coal getting where it is desirable to produce as little shattering as possible in order to produce as much lump coal as possible. By the use of special ingredients to reduce the density of the explosive and the development of special compositions it has been possible to produce permitted explosives that are particularly well adapted for the production of the maximum percentage of lump coal. These explosives which are now being introduced under the names of Rounkol and Rounkol No. 2, have been designed so that for equal bulk they are of about the same strength as Bobbinite or black powder, but owing to their low density, their efficiency, weight for weight, is much greater than that of the latter, so that in spite of the extra cost per pound and the necessity for the use of a detonator, the actual cost of explosives per ton of coal won should be less than before. Developments have also been made in connection with supplies of special types of black powder for special purposes, such as one of suitable burning speed for slate blasting, and one of particularly low density and high moisture resistance for use in tropical climates.

With rifle ammunition of the high velocity type, defects have been hitherto encountered due to metallic fouling and erosion. Both these defects have largely been met by incorporating a small proportion of an organo-metallic derivative of tin in the powder composition, and greatly increased accuracy in long range target competitions has resulted. Another interesting development during the year has been the marketing of a shotgun tracer cartridge, this being without doubt the most revolutionary improvement made in sporting cartridges for very many years. The tracing effect is obtained by the inclusion amongst the shot of a capsule containing a barium peroxide/magnesium mixture, and provision is made for this being ignited by the flame from the propellant powder. This tracer unit travels in the centre of the cone of shot and thus indicates by a brilliant spot of light the trajectory of the pellets. Resulting from the success of the military tracer bullets suggestions as to the desirability of such a sporting cartridge have frequently been made, but despite much work both in this country and abroad, the problem has, until this year, defied solution.

The Aliphatic Solvents Industry, 1931

Introduction of Many New Materials

Contributions to our knowledge concerning the different groups of aliphatic solvents, classified according to their chemical composition, are summarised in the following article.

THE world wide stagnation in trade which like a creeping paralysis has been spreading to every industry during the past two years, has latterly hindered the development of the solvents industry in this country. The effects of a crisis such as that through which all the world is now passing although first felt on the production and sales sides is soon, although not immediately, reflected by the slowing down of development schemes and of scientific research. As by their very nature neither new industrial developments on sound lines nor systematic and fundamental research can be hurried even under the most favourable conditions, the effects of any trade crisis is noticed in these fields long after the crisis has passed. The result of this lag is that a smaller number of articles of value now appear in the literature and there are fewer sound patents. It would be perhaps too optimistic to say that we are nearing the end of the present crisis but there are signs in many directions indicating that the worst has gone.

This country will no doubt share in any industrial revival now on its way and regain its prosperity, but the measure of this revival rests largely with the Government recently elected who have the power in their hands for this particular purpose and who can at least see that the manufacturers in this country have a fair opportunity of supplying the home markets. The manufacture of solvents in this country being a new industry, it is difficult to see how the test of abnormality of imports can be applied—the surer test in this case would be—ability to supply the full requirements of the country. It is to be hoped that the Government will seize every favourable opportunity of making wise commercial treaties with our Colonies and with other countries that are willing to trade with us on an equitable basis.

Properties of Solvents

The increasing use on an industrial scale of a number of chemical compounds that a few years ago were only produced and dealt with in small lots, has led to a more careful investigation of their toxicity, inflammability and so forth, and several articles in this field of some interest have been published during the year. The U.S. Bureau of Mines (*Bulletin* 279, 1931) has made a comprehensive survey of the limits of inflammability of a number of gases and vapours and has compiled a table of the inflammability limits expressed as percentages by volume in air. Among the substances dealt with are acetaldehyde, acetone, butyl acetate, ethyl acetate, alcohol, ether, methyl acetate and methanol.

Early in the year the U.S. Bureau of Mines issued a preliminary report on their investigation into the toxicity of methanol. They have not been able apparently to come to any definite conclusion as to how far methanol is toxic although they state in this report that there is no danger of poisoning from the reasonable use of methanol as anti-freeze. It would appear, however, that whilst occasional exposure to vapours of methanol is not dangerous, continued exposure undoubtedly is, and methanol whether crude or purified is poisonous when taken internally. Almost simultaneously with the above report was published the U.S.A. Government's decision to prohibit completely methanol as a denaturant.

An interesting description of the methods employed by the U.S. Bureau of Mines was given by Yant, Schrenk and Sayers in *Ind. Eng. Chem.*, 1931, **23**, 551. In a later article in *Ind. Eng. Chem.*, 1931, **23**, 931, C. McCord has summarised the results of a number of experiments on animals and has shown that either through absorption by the skin or through

inhalation of the vapour, small quantities of methanol quickly led to injury or death of the subjects treated, (monkeys, rabbits and rats). It was found that unit for unit, methanol is not less toxic when administered in the above ways than when swallowed. This, according to the author, would seem to call for protective measures for the safety of the public including industrial workers, which measures do not now exist. An article by McIntyre in the proceedings of the Society of Experimental Biology confirm these conclusions.

Through a series of experiments on animals with concentrations ranging from 1 in 50 to 1 in 10,000, Dr. Lederer (*Chem. et Ind.*, 1931, **25**, 72) has shown that the vapour of ethylene oxide is definitely toxic. The symptoms produced are, narcosis, followed by a drop in temperature, coma and finally death with the higher concentration. A report appeared in the German press of a case in which the inhalation of amyl acetate fumes from lacquers caused injury to the lungs of a worker and eventually death. This seems to be an extremely unusual case as the report of H.M. Chief Inspector of Factories and Workshops published last year concluded that amyl acetate was one of the less toxic solvents. The non-toxicity of diethyl phthalate has been shown experimentally by Blekendorfer and Templeton and the result of their work was published in the *Journal of the American Pharmaceutical Society*.

In an article on the choice of solvents and plasticisers for the manufacture of cellulose lacquers, T. Durrans (*Chem. Trade J.*, 1931, **88**, 53) summarises the composition and properties necessary in the solvent part of a good lacquer. Barbieri and Desmaroux have investigated the solubility of nitrocellulose in different alcohol ether mixtures (*Compt. rend.*, 1931, **192**, 1231.) and have found that the maximum solubility of nitrocellulose in alcohol-ether mixtures is obtained when the components are present in the solvent in equimolecular proportions. They have accordingly investigated the variation of solubility for mixtures of higher alcohols and their corresponding ethers and have found that it is closely connected with the stability of the oxonium salts of the ethers. A study of the compatibility of resins with nitrocellulose solvents has been made by H. Hoffman (*Ind. Eng. Chem.*, 1931, **23**, 127) from which it appears that resins may be divided into two groups, namely those which are more readily soluble in alcohol-type solvents and those which are more soluble in hydrocarbon-type solvents; most ordinary resins are not miscible under all conditions and with all solvents. The solubilities of oils and waxes were investigated by J. Poole (*Ind. Eng. Chem.*, 1931, **23**, 170.) who has collected together solubility-temperature data for the following solvents:—normal amyl alcohol, pentasol, butyl aldehyde, paraldehyde, ethyl chloro carbonates, ethylene dichloride, butyl formate, methyl ethyl ketone, two solvent naphthas and turpentine.

Methanol

The output of methanol in this country has undoubtedly diminished to a very considerable extent during the period under review. This is probably almost entirely due to general trade depression and as soon as industry in general revives a stimulus will be given to increased production. The high grade quality of British synthetic methanol affords another striking example of the efforts made by many chemical manufacturers in this country to turn out as current products, compounds of the highest grade of purity.

Continuing his work on the relation between the activity of the catalysts used for methanol synthesis and their chemical and physical structure, Professor Natta published in *Giornale di Chim. Ind. Appli.* 1931, **13**, 205, the results of his investigation of the relation between the coefficients of absorption of methanol catalysts for the gases present and their catalytic activity. The most important conclusions arrived at are that, chemical constitution being equal, the most active catalysts are those with the greatest absorption coefficients for carbon dioxide and carbon monoxide and that catalysts which have a high absorption coefficient for hydrogen are not suitable for methanol synthesis, since they promote secondary reactions such as the formation of methane.

A few patents relating to the synthesis of methanol have appeared during the year, but none of these present any special feature of interest, although U.S. Patent 1,797,569, Commercial Solvents Corporation, which describes a modification of the usual synthetic method and British Patent

346,658, Imperial Chemical Industries, which describes an ingenious method of purifying synthetic methanol are worthy of mention.

Although formaldehyde cannot be, strictly speaking, classed among the solvents, its manufacture is intimately bound up with that of methanol and in this connection two articles which appeared during the year on the production of formaldehyde by the catalytic oxidation of methanol with air deserve notice. The references are H. Adkins and W. Peterson, *J. Amer. Chem. Soc.*, 1931, **53**, 1512, and H. Kadowaki and M. Yamaji "Reports of the Osaka Imperial Industrial Research Laboratory" 1931. Nuber 19.

Ethyl Alcohol

Although ethyl alcohol principally in the form of methylated spirits is one of the most widely used solvents, its manufacture and distribution are so controlled in all countries by excise regulations that there is little scope for any radical departure from established practice. Since the closing down by the German Spirit Monopoly of the last German factory, which was producing ethyl alcohol from calcium carbide, reported in the press last summer, the whole of the world's ethyl alcohol supply with the exception of very small amounts from coke oven gases, is now produced by fermentation by a method which, however improved in technique is still fundamentally the same as that known to the Ancient Egyptians and probably still earlier in the world's history.

The Cie de Bethune and the Carbide and Carbon Chemicals Corporation are the only two concerns who are at present converting ethylene into alcohol on an industrial scale. The amount manufactured by the French company is only a very small fraction of the total French output, being about 15,000 hectolitres out of a total of 3,000,000, but according to a recent paper in "Facts about Sugar" the American Company are in a position to produce $3\frac{1}{2}$ to 4 million gallons of synthetic alcohol annually. An excellent description of the methods used by the Cie de Bethune was given by Sidersky in the *Bulletin de l'Association des Chimistes de Sucrierie et Distillerie* (1931, **48**, 163.) One very interesting patent in this field is that of the Distillers Company Limited, Joshua and Stanley, British Patent 360,492, which describes a method of absorbing ethylene in relatively dilute sulphuric acid under increased pressure.

According to articles in the American press which appeared early in the year, plans have been drawn up for the production of enormous amounts of industrial alcohol from the so-called "stabiliser" gases which are collected along with the crude gasoline at the Alberta oil wells. No further information is available as to how far this scheme has been proceeded with.

In an interesting paper which was read before the Royal society *Proc. Royal Soc.*, 1931, A.131, 533) G. Morgan and R. Taylor gave an account of the latest results of their investigations on the synthesis of ethyl alcohol and other compounds from water gas. By the use of a catalyst prepared from cobalt nitrate and zinc permanganate they were able to obtain a product containing about 10 per cent. of ethyl alcohol.

Anhydrous Alcohol

The important developments that have taken place during the past few years in the production of anhydrous alcohol have had for their principal object the manufacture of ethyl alcohol suitable for admixture with petrols to give motor fuels which are stable under all conditions. The methods of making absolute alcohol have now been so improved that it is stated that absolute alcohol can be produced as cheaply as rectified spirit of 95 per cent. strength. This should lead to the use of absolute alcohol as a substitute in place of rectified spirit in the solvent industries and this would offer many advantages which users of industrial alcohol might well consider. A summary of the most modern developments in the production of absolute alcohol was given by H. Guinot (*Chem. et Ind.*, 1931, **25**, 26.) A number of patents in this field have been granted during the year but for the most part these only deal with modifications of well known existing processes. An interesting process of a new type is described in British application 26620/30, Deutsche Gold und Silber Scheide Anstalt, in which the alcohol vapours are dehydrated by passage through a solution of a mixture of sodium and potassium acetates in absolute alcohol. This method is said to be particularly advantageous for small installations and the economy in steam consumption as compared with other processes is said to be considerable. An ingenious process for



*Left: Mr. C. A. Hill,
President of the Association
of British Chemical Manu-
facturers.*

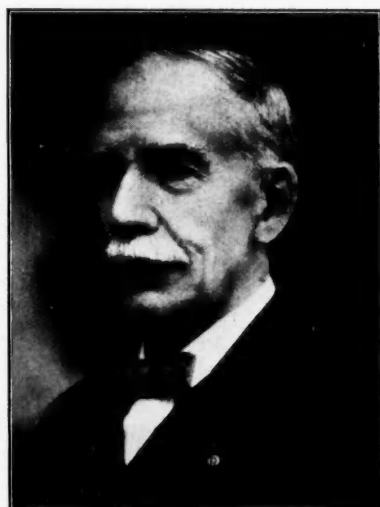
*Right: Dr. G. C. Clayton,
President of the Institute of
Chemistry.*



*Professor G. G.
Henderson, Pre-
sident of the
Chemical Society.*



*Dr. R. Seligman,
President of the
Institute of
Metals.*



*Left: Dr. J. T. Dunn, Pre-
sident of the Society of Public
Analysts.*

*Right: Mr. Victor Blagden,
President of the British
Chemical and Dyestuffs
Traders' Association.*



the direct production of absolute alcohol from the fermented wash is described in British Patent 350,473, Distilleries des Deux-Sevres.

Little of importance has appeared during the year in connection with the manufacture and use of ethyl ether although early in the year the Carbide and Chemical Corporation announced that they had produced this compound synthetically from an entirely new source and by an entirely new process.

Isopropyl Alcohol

Although the use of isopropyl alcohol as a substitute for ethyl alcohol seems to be spreading to a certain extent, particularly in the manufacture of cheap perfumery, practically nothing of importance has appeared recently in the literature in connection with this substance. The production of isopropyl together with butyl alcohol by fermentation is, however, claimed in British Patent Appl. No. 2761/31, J. Loughlin, as open to public inspection. In this process a special organism known as *Clostridium Americanum* is used.

Butyl Alcohol

The production of butyl alcohol by fermentation although now not carried out in Great Britain, seems to be extending in the Continental countries and during the year a new plant of considerable size has been put into operation at Strasbourg and appreciable amounts of fermentation butyl alcohol are also being imported here from Germany. Two articles of some interest on the chemistry of the butyl-acetone fermentation have been published in American journals during the year. In *Journal of Bacteriology*, 1931, 22, 79 Wynne has described the effect of both organic or inorganic acids on the fermentation of maize mash by the Weizmann organism under different conditions. Johnson, Peterson and Fred have studied the oxidation and reduction relations between substrate and products in the butyl-alcohol-acetone fermentation and have published the results of their investigations in *J. Biological Chem.*, 1931, 91, 569. Several patent applications in this field have also been granted or have become open to public inspection in the year but none of these contribute any marked developments in the art.

In spite of the handicaps of trade depression and the influx of butyl alcohol from the U.S.A. and the Continent, manufacture of butyl alcohol from ethyl alcohol is now well established in Great Britain and with improving conditions this new industry will undoubtedly be able to hold its own. With the exception of a number of patents, several of which have been granted only in countries outside Great Britain, nothing of importance has been published on the production of butyl alcohol from ethyl alcohol through crotonaldehyde. British Patent 353,071, British Industrial Solvents, Wiesler and Peake, which claims an improved method for the production of acetaldehyde from ethyl alcohol, British Patents 352,056, Boake and Townsend, and 353,413, Durrans and Lewis, which describe improvements in carrying out the aldol condensation and U.S. Patent 1,788,806, Commercial Solvents Corporation and Swallen, in which a new catalyst for the hydration of crotonaldehyde is disclosed, appear to be the most interesting of these patents. A method for the direct production of butyl alcohol from ethyl alcohol is claimed in British Patent Application 22588/30, Fuchs and Querfurth, which consists in passing a mixture of ethyl alcohol vapour and hydrogen over a special type of catalyst at relatively low temperatures. The formation of undesirable by-products and the loss of ethyl alcohol by decomposition which have been the principal drawbacks in the methods hitherto proposed for this condensation are said to be avoided.

Another organic synthesis parallel in its main lines with the production of butyl alcohol from ethyl alcohol via crotonaldehyde is the production of methyl isobutyl carbinol from acetone. Acetone is first condensed to diacetone alcohol, this is then dehydrated to mesityl oxide which in turn is hydrogenated to give methyl isobutyl carbinol. Methods for carrying out this synthesis on an industrial scale have been patented by the Distilleries des Deux-Sevres and by British Industrial Solvents. No important industrial development seems to have taken place in the production of higher alcohols from water gas although a number of patents, covering for the most part slight modifications in technique or plant, have appeared. An interesting article dealing with the chemical aspect of this synthesis was published by Natta and Strada in the *Giornale di Chim. Ind. Appl.*, 1931, 13, 317.

Higher Alcohols

The production of higher alcohols from the by-products of the petroleum industry falls naturally into two divisions. The first of these, that is the production of amyl derivatives from the pentane fraction of petrols, can only become of any importance industrially in those countries where petrol is found. No fresh developments seem to have occurred in America in this connection, but this method of manufacture seems to be attracting attention in other petrol producing countries, as Dobrianski and Gurewitsch in the *Russian Journal of the Petroleum Industry*, 1931, 15, 352, describe the result of their investigations on the possibility of obtaining amyl acetate from the pentane fraction of Krasnodar aviation petrol, and conclude that this could be carried out economically. The second method of obtaining alcohols from petrol by-products, that is the treatment of olefines obtained by cracking processes, has not yet become of great industrial importance, but it is highly probable that these gases may have to be seriously considered as sources of alcohols if vapour phase methods of cracking such as the Gyro process become adopted to any considerable extent. It is now in fact stated in the American Press that the Petroleum Chemical Corporation of New York is contemplating the erection of a large plant for the manufacture of secondary butyl alcohol from petroleum. Little else has appeared in the literature during the year with the exception of a number of patents, most of which, however, are of quite minor importance.

Before leaving the higher alcohols it would be as well to mention an interesting development that has taken place in the production of aliphatic alcohols of high molecular weight. A range of these products has been put on the market by the Deutsche Hydrierwerke A.G., of Berlin. These alcohols are manufactured from the corresponding carboxylic acids by the catalytic reduction of the carboxyl group using the process suggested by Professor Schrauth of Berlin and the alcohols made at present are those containing six or more carbon atoms to the molecule. These compounds may have important technical uses as they possess the property when dispersed with an emulsifying agent of mixing and holding in permanent suspension all hydrocarbons. The mixtures thus obtained should be valuable in combining the detergent properties of soaps with the solvent properties of the hydrocarbons. From a study of the patent literature it would appear that several other important industrial concerns such as the I.G. Farbenindustrie, the Bohme A.G. and the Du Pont de Nemours Company are also investigating compounds of this nature.

Acetic Acid

Although acetic acid cannot be properly classed as a solvent, its uses in the artificial silk industry and as a raw material or intermediate in the manufacture of other solvents are so important that a few words on it are not out of place. Acid produced by chemical synthesis is now the most important source of British made acetic acid and the establishment of this industry on a large scale and on an economic basis in this country is one of the most striking developments in organic chemical industry of the past few years. British Industrial Solvents Limited and Imperial Chemical Industries Limited share the honours of being the pioneers in this field.

For a general survey of the economic and technical developments affecting the manufacture of acetic acid since 1914 and the technology of the various processes used in its manufacture, the reader cannot do better than to refer to an article by E. Partridge which appeared this year in *Ind. Eng. Chem.*, 1931, 23, 482. Several patents covering improvements in the oxidation of acetaldehyde to acetic acid have been published of which the following seem worthy of mention:—British Patent 347,523, Holzverkohlungs Industrie, A.G., describes a process for the continuous production of acetic acid using an amount of oxygen in excess of that theoretically required for the reaction, the oxidation being carried out at temperatures between 55 and 80°C. The formation of per-compounds is avoided by bringing the gaseous mixture of acetaldehyde and the oxidising medium into contact with a large surface of dilute acetic acid. According to British Patent 350,874, C. F. Boehringer & Sons, acetaldehyde in the liquid phase preferable in solution in a chlorinated hydrocarbon is oxidised by a counter-current of oxygen. Plant for

use in the process consisting of five vessels in series is also described in the patent specification. In British Patent 359,878, Nippon Chissohrijo Kabushiki Kaisha, acetaldehyde is oxidised in a closed vessel into the lower part of which oxygen is bubbled whilst an excess pressure of inert gas such as nitrogen or carbon dioxide is maintained over the acetaldehyde. A number of patents have been granted to H. Dreyfus and to the British Celanese for the synthesis of acetic acid from water gas or from a mixture of water gas with methyl alcohol or methyl esters, but it is very doubtful whether this method of synthesis has yet been developed sufficiently for use on an industrial scale.

A new method for the synthesis of acetic acid is claimed in British Patent 355,866, Bataafsche Petroleum Maat in which ethylene and caustic alkalis are caused to combine under high pressures and temperatures in the presence of catalysts. Several patents for the concentration of dilute acetic acid for the recovery of acetic acid used in cellulose acetate manufacture have appeared during the year but these for the most part are only slight modifications of well known existing methods.

Ketones

Unfavourable conditions in all industries in which acetone finds an outlet have effected the manufacture of this compound throughout the world and large quantities have been dumped in this country from the U.S.A. and the Continent. From the technical point of view also very few developments of any importance have taken place and the year has been a period of "marking time." The use of silica gel as a catalyst for the production of acetone from acetic acid was studied by Mitchell and Reid and the results of their investigation which shows that only mediocre yields could be obtained by this method were published in the *J. Amer. Chem. Soc.*, 1931, **53**, 338. An ingenious method for the production of acetone from cellulose acetate precipitation liquors is described in German Application W. 77680, Wacker. Ges. fur Elektrochemische Industrie. The vapours from these liquors are passed directly without purification over cerium carbonate on a pumice support and a high degree of conversion is said to be obtained. A number of modifications of the known methods of producing acetone from alcohol or acetylene and steam consisting principally in the use of special catalysts have been patented, but none of these constitute any great advance in the art. Processes for the production of acetone from isopropyl alcohol have been patented by the Bataafsche Petroleum Maat, British Patent 339,491 and by the Deutsche Gold und Silber Scheide Anstalt, British Patent 359,430. An acetone derivative obtained by treating acetone with ozone and said to be useful as a bleaching agent is described in German Patent 495,021, Chemische Fabrik von Heyden.

Few articles and patents of interest have appeared in connection with the higher ketones. In an article in the *Amer. Paint J.*, 1931, **15**, 45, Park and Kofmann have given a systematic study of the aliphatic ketones, acetone, methyl ethyl ketone, methyl butyl ketone and methyl amyl ketone from the point of view of their properties as solvents. The ketones studied are the principal constituents of the "acetone oils" obtained in wood distillation. According to British Patent 347,593, Holzverkohlungs Industrie A.G. ketones can be produced from alcohols or their derivatives such as aldehydes and esters by the action of water vapour at high temperatures in the presence of catalysts containing oxides or carbonates of cobalt and nickel. Examples are given of the production of methyl ethyl ketone from crude ethyl propionate and of dipropyl ketone from butyraldehyde. In British Patent 346,822, Société des Brevets Etrangers Lefranc, for the production of higher homologues of acetone it is claimed that higher yields and purer products can be obtained if the alkaline earth salts of the aliphatic acids used are distilled in the presence of calcium hydrate.

Esters

The manufacture of esters has suffered less than most chemical industries from the abnormal world conditions and another bright spot in the picture is that practically all the esters used in this country are British made and to a very considerable extent manufactured from raw materials of British origin. According to American trade journals the Du Pont de Nemours Company have recently put on the market a new ester solvent known as "34 solvent." It is

composed mainly of ethyl butyrate and apart from its decidedly unpleasant smell of which, however, it leaves no trace on evaporation, it is said to possess all the properties of a valuable solvent. It has a boiling range of 110-123°C., is insoluble in water and has a specific gravity of 0.872 to 0.878 at 20°C.

A very interesting article on the aceto-acetic ester condensation was published by Snell and McElvain in the *J. Amer. Chem. Soc.*, 1931, **53**, 2310. This is more particularly important as a contribution to pure chemistry, but the light thrown on the reaction between sodium and ethyl acetate in the presence of alcohol may have some importance later from an industrial point of view.

Quite a number of patents have been published in connection with the manufacture of esters and of these several present interesting features. British Patent 352,647, Holzverkohlungs Industrie, A.G., describes a process for the continuous production of ethyl esters directly from fermented washes and a further claim of the patent is for the use of crude pyroligneous acid in conjunction with dilute alcohol vapours for the manufacture of ethyl acetate. Another method for utilising dilute aliphatic acids in ester manufacture is described in British Application 14124/31, Usines de Melle. The acids are extracted from the dilute aqueous solutions by a solvent slightly soluble in water preferably with the alcohol to be esterified. Claims are made for the advantage of the process in producing esters from the dilute solutions of acetic acid obtained in the artificial silk industry. Quite another type of process is described in U.S. Patents 1,817,898 and 1,817,899, Commercial Solvents Corporation, as in these patents it is claimed that esters can be obtained from the primary alcohols by passing them in the vapour form over a catalyst consisting of uranyl carbonate with either silver carbonate or metallic silver. An alternative procedure is to pass the alcohol vapours successfully over a series of catalysts consisting of barium uranyl carbonate, silver carbonate, copper carbonate and alumina. U.S. Patent 1,781,050, Carbide & Carbon Chemical Corporation, describes the production of ethyl esters by the interaction of diethyl sulphate with the sodium salt of the acid to be esterified and a somewhat similar process is described in British Application 17473/31, I.G. Farbenindustrie.

Production of Esters from Olefines

The production of esters directly from olefines is claimed in British Application 1886/31, Standard Oil Development Company in which esters are obtained by continually passing a stream of olefine in a counter-current of anhydrous liquid organic acid in presence of a suitable acid catalyst. Two extremely interesting patents relating to the manufacture of esters of lactic acid have been granted to Imperial Chemical Industries, British Patent 341,961 and 346,486. In the former lactic acid esters are obtained by condensing acetaldehyde with gaseous hydrocyanic acid in the presence of a lactic ester. No separation of the acetaldehyde cyanhydrin is necessary. In the second named a process of alcoholysis is described which is particularly adapted to lactic esters. Another patent dealing with alcoholysis is British Patent 341,730, Imperial Chemical Industries, filed as a communication from the Du Pont de Nemours Company. U.S. Patent 1,790,262, L. Christman, also deals with a process of forming lactic esters from nitriles, the esterification being carried out in an inert solvent by hydrochloric acid generated *in situ*. Another process making use of nitriles is disclosed in British Patent 352,802, I. G. Farbenindustrie, for the manufacture of chlor-propionic esters and according to British Patent 351,518 of the same patentees, esters of chlor propionic acid can be converted into acrylic esters by heating them in the absence of moisture with an inorganic non-volatile catalyst of an acid reaction.

An improved method of preparing esters of glycollic acid is described in British Patent 337,609, I. G. Farbenindustrie, which consists in heating the dry alkaline salts of chlor acetic acid, free from water of crystallisation, with aliphatic alcohols at pressures above atmospheric. A process for the production of esters of poly-basic acid from alcohols obtained from oil cracking gases is described in U.S. Patent 1,815,878, Van Schaack. The manufacture of alkyl phosphoric esters is described in U.S. Patent 1,799,349, Commercial Solvents Corporation, and according to this specification these esters can be obtained by the interaction of aluminium alkoxide with phosphorus oxy-chloride.

Some Problems of National Importance

The Work of the Department of Scientific and Industrial Research

This article contains a brief review of some chemical researches in progress in research stations maintained by the Department of Scientific and Industrial Research, but no reference is made to the chemical investigations undertaken by the Research Associations under the Department's scheme for promoting research in industry.

THE work of the corrosion section of the Chemical Research Laboratory deals with zinc, iron, steel, copper and magnesium alloys; part is devoted to the elucidation of the mechanism of corrosion and part to technical problems of protection. The measurement of corrosion by the oxygen-absorption method developed for zinc has now been applied to iron and mild steel. Hydrogen gas was evolved in potassium chloride solutions and the proportion of the total corrosion due to this type of action was considerable in all solutions stronger than N/1,000. The results have shown that the characteristic distribution of corrosion over completely immersed horizontal specimens was determined neither by the initial air-formed film nor by any differential aeration that could occur in stagnant solutions, but mainly by the distribution of a particular form of precipitated corrosion product. The deposits of rust which are usually supposed to behave as oxygen screens and stimulate corrosion by differential aeration do not so behave in any potassium chloride solution stronger than N/1,000; the rust was found to consist almost entirely of mixtures of ferric oxide and stable magnetic oxide. In very weak solutions in the presence of oxygen the rust was usually built up into coherent mounds which acted as oxygen screens but gradually reduced corrosion rates.

Exploratory work on the protection of magnesium alloys has involved an examination of more than five hundred coatings from which twelve of the more promising have been selected for further trials. Among the selected coatings were those containing a chromium basis derived from either chromic acid or an alkali chromate. These experiments were followed by the important discovery that protective films of selenium are deposited when sheets of light magnesium alloys are immersed in acidified solutions of selenium compounds. Such selenium films exhibit a noteworthy resistance to intermittent spraying with salt water. The optimum conditions for the deposition of the films on alloys in general use are being investigated in detail.

Canning Industry

A small canning laboratory was equipped in 1927 at the Low Temperature Research Station, Cambridge, for the study of the fundamental problem with which the canning industry is confronted, namely, the corrosion of the tin-plate container. Work has been in progress on the corrosion of iron, tin and tin-plate by fruit acids and other substances present in canned fruits. The importance of studying the influence of a particular factor or substance under carefully controlled conditions, and over a wide range of acidity, has been increasingly evident. For example, traces of sulphur dioxide accelerate the corrosion of iron at high acidity, but actually retard it at low, whilst gelatin and other inhibitors of acid corrosion may exert a powerful influence at high acidity but show little or none at low. The tin coating of tin-plate is rapidly removed at acidities corresponding to those of the less acid fruits but is relatively free from attack in media comparable with sour fruits. This discovery helps materially towards an explanation of the formation of hydrogen-swells and perforations, to which losses with certain important products are due, and has also led to the practical and beneficial expedient of adding a small percentage of citric acid in canning fruits of low acidity.

High Pressure Chemistry

A general investigation of high-pressure synthesis initiated in the Chemical Research Laboratory in 1926, has been continued. A diversity of experience in regard to the formation of ethyl alcohol as a product of high pressure synthesis from carbon monoxide and hydrogen having been revealed, a new series of experiments has been made. The results obtained confirm the earlier observations on the production of ethyl alcohol. Several mixed catalysts have been found to induce this synthesis and the presence of ethyl alcohol in such condensations is now placed beyond reasonable doubt. Further

examination of the complex organic products of carbon monoxide-hydrogen condensation has now indicated the presence of acetone, methyl ethyl ketone, ethylidene dimethyl ether and propylidene dimethyl ether.

Low Temperature Tars

Investigations of tars obtained by distillation of coals at low temperatures have been continued at the Chemical Research Laboratory and the Fuel Research Station with promising results. Laboratory experiments at the Fuel Research Station have shown that a large proportion of the tar can be converted into true oils and motor spirit with an absence of pitch, and that this applies not only to "low temperature" tars, but also to some fractions of gas and coke-oven tars. Hydrogenation at high temperatures and pressures in the presence of catalysts has been studied by two series of experiments dealing (1) with the fraction boiling above 230°C. and (2) with the whole crude wet tar. Laboratory experiments with the fraction boiling above 230°C. have shown that the largest conversion to spirit is obtained by using a catalyst consisting of active charcoal or silica gel, impregnated with ammonium paramolybdate, with sulphur added separately. Treatment of the whole crude wet tar using the same catalyst has shown that the volume of oil obtained is the same as the volume of tar treated. It will not be possible to judge of the costs, and, therefore, of the economic possibilities of this process until the experiments have been repeated on a larger scale.

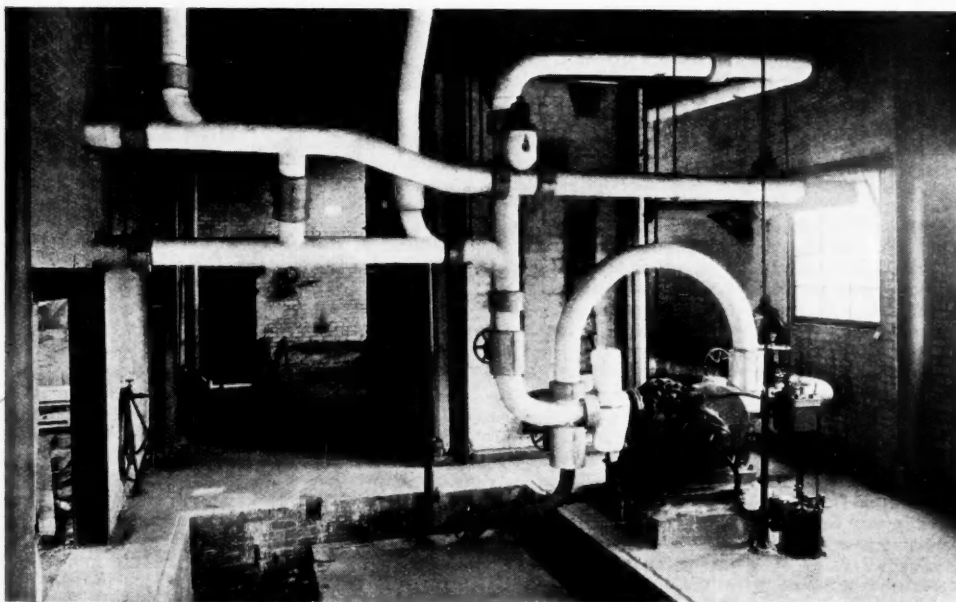
At the Chemical Research Laboratory a systematic examination of the anthracene fraction of low temperature tar has confirmed the presence in this material of anthracene and its homologues. In addition to the mono-, di- and tri-methyl derivatives of anthracene previously studied the new 2:3:6:7-tetramethylanthracene and the corresponding tetramethyl-anthraquinone have been synthesized and the former identified as a constituent of low temperature tar. Among the higher phenols of low temperature tar identified by means of phenyl-carbimide and *p*-xenylcarbimide are the following: *o*-homocatechol, *isohomocatechol*, resorcinol and quinol. The comparative study of industrial low temperature tars has been pursued and some seven and eight of these tars have been separated into their main constituents.

Hydrogenation of Coal

The main line of work on the hydrogenation of coal in progress at the Fuel Research Station has been directed to the study of the action of various catalysts, a large number of which have been examined. A report (*Fuel Research Technical Paper No. 29*) has been published giving details of experiments showing how the caking properties of a coal can be modified by treatment with hydrogen under pressure. The products obtained by partial hydrogenation of coal are also being examined in connection with the study of the chemical constitution of coal. Partially hydrogenated products from Arley and Tydraw coals have been subjected to benzene-pressure extraction and the extracts each separated into fractions following the scheme put forward by W. A. Bone, L. Horton and L. J. Tei (*Proc. Roy. Soc. A.* 1928, 120, 523). Comparison of the yields of the fraction indicates that the increased caking capacity of partially hydrogenated coal is due to increase in fraction IV.

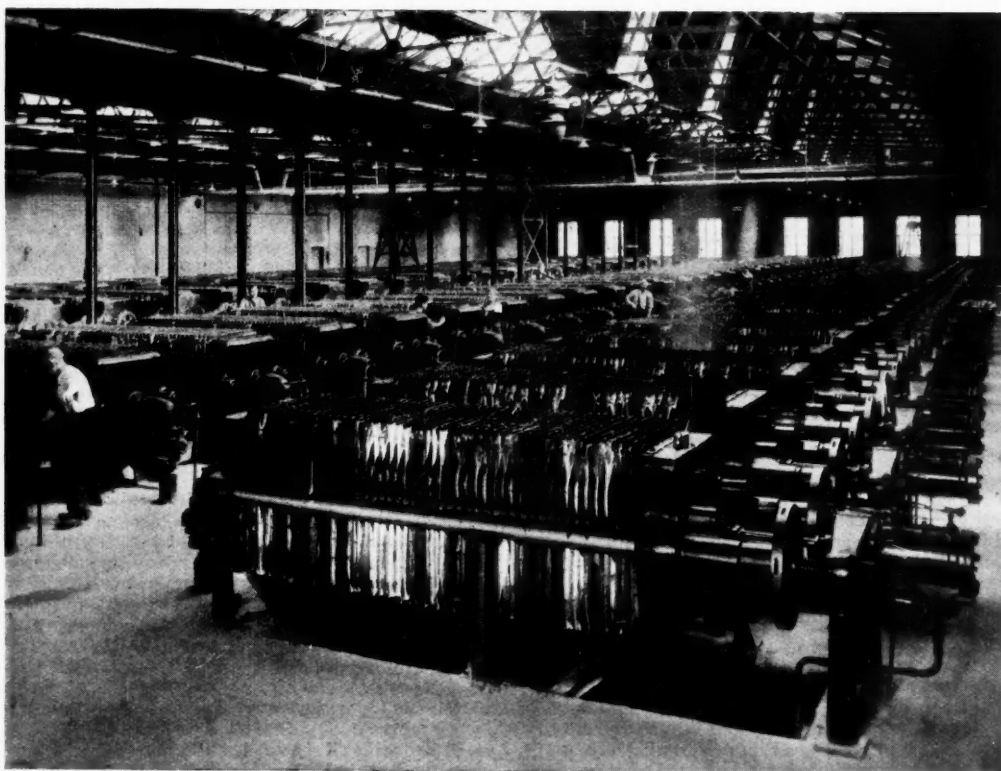
Synthetic Resins

Research on formaldehyde condensations with acetone and its homologues in alkaline media has been continued. The results show that, as the homologous series is ascended, larger quantities of alkali and more prolonged heating are required in order to produce resins. Intermediate stages in these condensations have been identified in the case of acetone, methyl ethyl ketone and also diethyl ketone. Owing to the slow heat-hardening character of the acetone resin, attempts to use it as a moulding material have failed. More success



THE ABSORBER ROOM OF A KESTNER PATENT OIL CIRCULATION HEATING PLANT, CAPACITY
3,200,000 B.T.H.U PER HOUR.

(KESTNER EVAPORATOR AND ENGINEERING CO., LTD.).



THE FILTER HOUSE, BRADFORD SEWAGE WORKS: PART OF THE PLANT FOR THE RECOVERY
OF GREASE FROM WOOL WASHING WASTES.

has attended the utilisation of the resin as a varnish, either alone or incorporated with colophony.

Condensations of phenol and formaldehyde with glycerol and polybasic acids or anhydrides have yielded resins designed to eliminate objectionable "tracking," the products combining the properties of "bakelite" and "glyptal." In addition to further experiments on pure phenolic substances, involving semi-works scale processes and in which alkylamines have proved efficacious catalysts, investigations have been made of formaldehyde condensations with phenolic fractions of low temperature tar. These resin preparations when tested either on single impregnated sheets or in laminated boards have breakdown voltages at least equal to those of trade resins and of resins made under comparable laboratory conditions from pure phenol. Soluble non-hardening resins of Novolak type accompany hardening resins in formaldehyde condensations with crude phenols. Work is now in progress with the object of cheapening the methods of production. The results represent a technical advance of some importance in view of the large amount of crude phenols which would become available if low temperature distillation of coal were practised generally.

Water Pollution Research

During the past three years experiments have been in progress on behalf of the Water Pollution Research Board at the Chemical Research Laboratory on the base-exchange or zeolite process of water softening. The exchange values of typical commercial materials have been determined at different rates of flow of water and after various methods of regeneration. In connection with an investigation of the effects of variations in the composition of the water to be softened, experiments made with solutions of magnesium sulphate in distilled water have shown that the amounts of magnesium removed by base-exchange materials appear to be less as compared with calcium than those corresponding with equivalent proportions of the two metals. It has been shown that base-exchange materials may be regenerated by treatment with sea-water in place of solutions of common salt ordinarily employed, but, owing to the presence of calcium and magnesium in solution, sea-water is not so effective as a solution of common salt containing sodium ions in the same concentration. The interesting observation has been made that base-exchange materials remove from water such undesirable metals as copper, lead, tin and zinc which may be present in solution.

Chemotherapy

Investigations are proceeding at the Chemical Research Laboratory in collaboration with the Chemotherapy Committee of the Medical Research Council in the preparation of materials likely to be of therapeutic value. Attention has been directed mainly to the analogues of the Bayer 205 or Fournau 309 type containing carbazole, fluorene and fluorenone nuclei. Certain members of a new group of organic arsenicals derived from *p*-arsanilic acid have shown considerable therapeutic activity and compare favourably with the American drug tryparsamide. The preparation of this series is being continued and experiments are also in progress on another new series of arsenicals derived from fluorene.

Building Research

The need for satisfactory means of determining the amounts of hydrated and unhydrated free lime in cements and other building materials is frequently encountered. The Building Research Station has recently completed a comprehensive review of the methods now available. Three types of method are found to be of value and four actual methods, namely, two modifications of the glycerol extraction method, a lime solution extraction method and a calorimetric method, have been studied in detail. The last two are new, and one modification of the glycerol method embodies fresh variations. The glycerol methods are shown to be applicable to unhydrated cements, to sand-lime bricks and also to hydraulic lime. The calorimetric method was devised for hydrated cements, and may also be used for mortars and concrete. The lime solution extraction method is restricted in its application to lime mortars and hydrated limes.

The weathering of matured building stones is of considerable practical interest to the building industry and presents

many problems for solution by the chemist. A detailed survey of the general subject carried out at the Building Research Station has included a study of the chemical phenomena associated with weathering. In particular, investigations of the action of acid sulphur gases upon limestone and of the disintegration caused by the crystallisation of salts in the pores of building materials have given results of immediate practical value. The large scale experiment on "stone preservatives" which was initiated in 1926 has indicated that none of the methods of treatment appears to have protected the stone from weathering agents. The advantages of washing in keeping stonework clean and in retarding decay and also the danger of using alkalis or other chemicals for cleaning have been demonstrated.

Storage of Fruit

In the past few years the study of the chemistry of the apple in relation to its storage-life, which has been carried out at the Imperial College of Science and Technology on behalf of the Food Investigation Board, has proceeded along two main lines, namely, a detailed study of the chemical changes during storage life and a survey of the variations in composition at gathering of fruit from different sources and of different varieties. The investigation of chemical change in store has yielded some definite results, such as the inverse relation between the length of storage-life and both nitrogen-content and rate of loss of sugar and acid in respiration, and also the increased rate of loss of acid with the onset of internal breakdown. Continuation of the study of the effect of time of gathering on the storage life of the apple suggests that if prematurely gathered fruit is placed in cold store, the starch present gives rise mainly to reducing sugars, with only a very little sucrose. Quantitative determination of starch in the growing fruit has shown that a considerable amount of easily hydrolysable polysaccharide, other than starch and pectin, is formed during growth. A small amount of such a substance, which yields arabinose as one of its products of hydrolysis, has been isolated.

Storage of Meat

When efforts are made to preserve meat it is frequently found that the limiting factor is the fat, which becomes rancid or tainted while the lean or muscle is still in good condition. Scientific analysis carried out at the Low Temperature Research Station, Cambridge, has shown that fat-taints may arise from oxidation or from the action of micro-organisms. It has now been found that, in the case of frozen lamb and chilled beef, certain micro-organisms are the most important cause of fat-taint, while in the case of frozen bacon, oxidation is the principal factor. Where micro-organisms are the cause of taint, a high free acidity is developed. A quantitative test for the detection of oxidation in fats has been described together with a technique for quantitative application of the Kreis reaction (*Proc. Roy. Soc. (B)*, 1931, 108, 175). Oxidation is accelerated sensibly by weak artificial light while exposure for a few minutes is sufficient to produce rancidity in the superficial layers. The reaction is autocatalytic and even brief exposure to light accelerates the subsequent oxidation.

Fish Oils and Fats

An investigation of the activity of lipase in the livers of various species of fish has been commenced at the Torry Research Station with the objects (1) of determining the retrogressive changes which take place in liver fat after the death of the fish; and (2) of studying the metabolism of fat in living fish, in which the liver-enzymes play so fundamental a part. The fats from a large variety of species are being exhaustively analysed and it has been shown that there are fundamental differences of composition between the fats of marine species on the one hand and of fresh water species on the other. The unsaponifiable constituents of fish oils are also being studied jointly with the Department of Organic Chemistry, University of Liverpool. Of particular interest is a characteristic pigment, which occurs in the liver of the angler fish (*Lophius piscatorius*) and which possess carotinoid properties. No other fish's liver yet examined contains this pigment, and its study may throw some light on the conversion of carotene into vitamin A in the liver of animals receiving the former in their diet.

Conditions of the Heavy Chemical Industry in 1931

By P. Parrish, F.I.C., M.I.Chem.E., M.I.Gas E.

Mr. Parrish once more reviews the general conditions in the heavy chemical industry, dealing more especially with those problems which affect by-products and synthetic ammonia, sulphuric acid, phosphate and superphosphate, and the hydrogenation of coal.

IN concluding last year's review, it was remarked that trade depression and unemployment in this country were due more to the instability of gold as a standard of value than to over-production. Matters failed to improve during the early months of 1931; indeed, they grew worse. Confidence was rudely shaken, and the British nation suddenly found itself on the verge of a financial downfall in August and September last. It would be inappropriate here to discuss what caused the crisis, but national extravagance, and the neglect of the interests of industry, contributed their quota. That the heavy chemical industry, in common with other industries, has suffered, is obvious from the fact that for the first nine months of this year, exports have dropped £4,123,201, and imports have fallen £1,091,692, below the figures for the corresponding period of 1930. The National Government is tackling the problem in a commendable way, but many of the salient factors of the crisis still await solution.

Synthetic Ammonia

The number of new patents relating to the production of synthetic ammonia has again diminished. Indeed, nothing of outstanding importance can be reported in this connection. Much work of a consolidating nature has been undertaken. The various well-known processes, differing according to geographical position, availability and character of raw materials, power, etc., are now firmly established. Although improvements in plants and methods continue to be made, these do not appear so significant as when the industry was in active development.

Despite the over-production of ammonium sulphate, synthetic ammonia plants are in course of erection in Belgium, Hungary and Russia. The Mont Cenis process, working at comparatively low temperatures and pressures, utilising coke oven gas, has proved to be reliable and sound, and it is certain that if circumstances warranted further extension of synthetic ammonia plants in this country, one could not lightly disregard the claims of this process, which can produce ammonia from coke oven gas at less than £6 per metric ton of ammonia a year. In other words, ammonia costs, per unit of 22.0 lb., about 1s. 2½d. This is a figure that ammonia experts, by-product and synthetic, should commit to memory.

By effecting the reactions between water gas and steam in successive stages, the Du Pont Ammonia Corporation claim that hydrogen is produced with a minimum formation of methane and carbon dioxide. The steam is mixed with approximately one-third of the theoretical quantity of water gas, to reduce the temperature again to 420° C., and once more passed over the catalyst. If desired, the operation may be repeated a third time. A patent (U.S. Patent 1,765,534) has been granted to the Chemical Engineering Corporation, for purification of the gas mixture prior to synthesis. Approximately 0.2 per cent. of ammonia is added to the mixture before compression, whereby the aqueous condensate obtained on compression contains all the catalyst poison present in the gases, e.g., chlorine, sulphur and phosphorus compounds.

In the production of phosphoric acid, calcium sulphate sludge remains. This is being converted by the large fertiliser works in Belgium, Germany, Holland, and Italy, into ammonium sulphate, by reaction with ammonium carbonate. The resulting residue—calcium carbonate—constitutes a raw material for reaction with nitric acid (produced by the oxidation of ammonia by the Ostwald process), in the formation of either calcium nitrate or nitro chalk.

The failure to renew the international nitrogen agreement is a matter of concern to by-product and synthetic ammonia manufacturers in Great Britain, Germany, France, Belgium, Holland, Italy, Poland and Czecho-Slovakia. Had an agreement been reached, world prices would doubtless have been maintained. It is reported that the representatives of the Chilean industry demanded a far greater proportion of the world trade in general, and also that they claimed a share of the German market four or five times greater than the pro-

portion they secured in 1930-31. In a final attempt to reconcile the views of the parties concerned, it is alleged that negotiations broke down by reason of the refusal of the Chilean representatives to engage in further negotiations with the representatives of the German nitrogen industry. It is to be regretted that in an attempt to consolidate still further their position, the Chilean representatives should have been largely responsible for a vigorous price war, as a result of which all nitrogenous fertiliser manufacturers must inevitably suffer.

Scientific Utilisation of Coal

Further advance has been made with the hydrogenation of coal, high and low temperature tars, and creosote oil. Progress has already reached a stage at Billingham sufficient to admit of the hydrogenation of 15 tons or more a day of bituminous coal. With clean, dry, coal, so treated, some 60 per cent. by weight is recovered as refined petrol.

The cost of making petrol from tars depends on the price at which tars are available. At £2 per ton, petrol can be made at 7d. per gallon. If tars were available at or about this price, they would constitute a suitable raw material for hydrogenation. A plant built specially for the hydrogenation of tar would not be suitable for the hydrogenation of coal, without considerable alteration. It is estimated, by K. Gordon, that £8,000,000 is required for hydrogenation plant capable of treating a thousand tons of coal per day, and producing 210,000 tons of petrol per annum. It is not recommended that smaller plant should be installed, as it would not constitute an economic unit. An estimate of the cost of petrol made in a plant of the foregoing size, would be 7d. per gallon, compared with the present market price of 2½d. per gallon.

The chemical and engineering aspects of the hydrogenation of coal and other suitable raw materials no longer present any difficulty. The problem that remains is essentially an economic and commercial one, bound up with the uncertainty of the 8d. tax, which constitutes a subsidy of the first order to home producers of petroleum and benzole. Meanwhile, Germany has erected large-scale plant for the treatment of partially dried and briquetted lignite in power gas producers, affording gas for power purposes, and also a nitrogen-containing gas for the synthetic ammonia process. Scrubbing the producer gas to render it suitable for power purposes affords large quantities of tar, the distillation of which yields certain suitable fractions for hydrogenation. Similarly, tars from the low temperature carbonisation of lignite and bituminous coal, are hydrogenated, to yield naphtha, which can be utilised as motor spirit.

Sulphuric Acid

The demand for sulphuric acid has diminished rapidly during the last twelve months. Hitherto, the major part of this mineral acid was absorbed in the manufacture of ammonium sulphate and calcium superphosphate. The low prices obtaining for sulphate of ammonia since July last have led to the disposal or destruction of gas liquor in many and varied ways, which have not called for the use of sulphuric acid.

Sulphuric acid manufacturers have had a very lean time, and the prospects of emergence from the crisis are not too hopeful. An improvement in world trade will have a favourable influence, but it cannot be denied that fundamental changes are being wrought. Certain elements in the structure of the heavy chemical industry are undergoing a definite metamorphosis. Indifference to these change can only lead to one inevitable penalty, namely, loss of trade or perhaps gradual supercession. Like the acid manufacturers, various companies mining pyrites have suffered no less severely from an absence of demand for their raw material. Some of the mining companies have turned their attention, as briefly indicated last year, to the recovery of sulphur from pyrites. A number of processes have been patented, but one which holds out considerable promise involves the treatment of pyrites

with coke in a blast furnace, where, under suitable conditions of temperature and air regulation, the products of combustion and reduction emerge containing sulphur in the sublimed form, which, for the most part, can be collected in a hot electrostatic precipitator.

Developments in the flash roasting of iron pyrites have been made by Horace Freeman. The best flash method of combustion of pyrites has been found to be by injection with air downwardly into a vertical combustion chamber. Only part of the air needed for complete combustion is used to convey the pyrites: the remainder is caused to ascend the chamber in counter-current to the descending pyrites. The utilisation of the exothermic heat developed also constitutes a fascinating study, but no further inroads have been made in this country by the contact process, despite the existence of vanadium as a catalyst.

With the Gaillard-Parrish liquid phase sulphuric acid plants, it is possible, with a special provision, to produce from 30 to 60 per cent. of the total make of acid as C.O.V.—94 per cent. H_2SO_4 . This fuel-less type of concentrator has attractive features, and must arrest the suppression of the chamber system. To render possible the production of C.O.V. without extra fuel, without labour, and without the necessity for auxiliary plant for arresting the acid mist and fumes, with a material reduction in costs of repairs and maintenance, and in initial capital expenditure, represents a progressive step in technique of the first order. Where there is only a partial demand for C.O.V., 94 per cent. H_2SO_4 , it still pays handsomely to produce strong acid. Any surplus strong acid can be used to raise the strength of the circulating acid to the Gay Lussac towers. The last traces of SO_2 are vigorously absorbed in the stronger nitrous acid, because there is a virtual increase of surface. Apart from this, the absorption of oxides of nitrogen is appreciably more effective, and a sensible economy in the consumption of nitric acid, or its equivalent, is effected.

Experience in the operation of the Gaillard-Parrish system during the last twelve months, has revealed that intensive production is largely dependent (a) on the nitrosity of the nitrous acid in circulation, (b) on the extent to which the dispersed chamber acid is cooled, and (c) on the fineness of the dispersed chamber acid. Factors (a) and (b) are of primary importance. With a nitrosity of 50 oz. $NaNO_3$ /c.ft. of acid, it is possible to work with spent oxide on a space-time factor of less than 3 cubic feet/lb. sulphur/per day. Such performance is related to reaction temperatures in the first chamber not exceeding $68^\circ C.$, and in the second not exceeding $60^\circ C.$ The surface temperature of the lead of the first chamber does not exceed $30^\circ C.$ at the top portion, and $42-45^\circ C.$ at the bottom portion of the chamber. The temperatures in the second chamber are respectively $30^\circ C.$ at the top, and $38^\circ C.$ at the bottom. With such phenomenally low temperatures, wear and tear of the lead is negligible, and the development of fatigue is sensibly retarded.

By-Product Ammonia

The failure to renew the international nitrogen agreement has had an adverse effect on the by-product ammonia industry in this country. A reduction of about 50s. per ton in the price of sulphate of ammonia cannot be contemplated with equanimity by the average gas undertaking or coke oven works. But the resources of this industry are not yet exhausted. Economic pressure can only result in combined effort in the establishment of centrally-situated chemical works, the necessity for which has long been foreseen, and in a modern attack of the relevant problems.

Where ammonium bicarbonate is crystallised under special conditions, and steps are taken to eliminate almost completely the moisture content, a product which has little tendency to dissociate can be made. It seems certain that extended uses for such a product will be available before long. Each town produces certain organic residues, which, if suitably treated and admixed with ammonium bicarbonate, would constitute a fertiliser of the first order. The features of such a fertiliser, apart from its nitrogenous value, would be lightness and porosity, and it would be peculiarly suitable for application to many of the heavy soils which abound throughout the country.

Centrally situated chemical works, dealing with by-product ammonia, must not lose sight of the fact that they are admirably placed for producing nitric acid, calcium nitrate from

the lime residues of their sulphate of ammonia plant, nitro chalk, and even nitrate of ammonia. Developed on a sufficiently large scale, with economic plant, there is nothing to prevent these products being produced on a competitive basis.

Various methods have, from time to time, been put forward for the production of ammonium sulphate directly from gaseous sulphur dioxide, ammonia and water. None of these processes has met with real commercial success. Two new proposals are, however, worthy of note. According to S. I. Volfkovich and D. L. Tzirlin (*J. Chem. Ind., Russia*, 1929-6-1323), ammonium sulphite in aqueous solution can be completely and quickly oxidised by air in towers filled with silica gel or quartzite. The General Chemical Co. proposes to introduce SO_2 into ammoniacal solution, so as to give a solution containing at least two molecules of bisulphite per molecule of sulphite. This solution is heated to $200^\circ C.$ under a pressure of 700 lb. per sq. inch, after which it is claimed that sulphur is separated, and the solution can be evaporated for recovery of the salt.

Alkali

The established processes still continue to operate successfully. The ammonia-soda process is a highly organised technical operation, and is peculiarly efficient. Improvements have been made during the year, both in the construction and working of mercury and diaphragm cells. Indeed, the latter have been modified in important respects, as compared with five or six years ago. If salt were more widely available, it is certain that the by-product ammonia industry would not hesitate to embark on the production of soda ash by the ammonia-soda process. Concentrated gas liquor affords a suitable starting point. Fortunately, certain sodium chloride waste liquors are becoming increasingly available, and a study of these merits attention, for the purpose foreshadowed. Increasing use is also being made of concentrated hypochlorite, and extended applications of liquid chlorine can be witnessed in many parts of the world, for the purification of water, and for the treatment of circulating water, to arrest growths of algae.

Phosphoric Acid

At least two works, one in Holland and another in Canada, are now producing phosphoric acid by improved processes, where hemihydrate crystals are permitted to deposit on old crystals, and where filtration difficulties have been minimised considerably. Now that the chamber process can yield 30 to 50 per cent. of its make as C.O.V., at a negligible expense, it is certain that the tendency in the future will be to produce phosphoric acid containing 32 per cent. P_2O_5 , by using stronger sulphuric acid than has hitherto been customary. Plants for the concentration of phosphoric acid by the submerged combustion method have been erected on the Continent, and bid fair to solve this rather intractable problem of evaporation, with its attendant difficulties, due to the deposition of pyro and meta phosphates—largely the products of surface local heating.

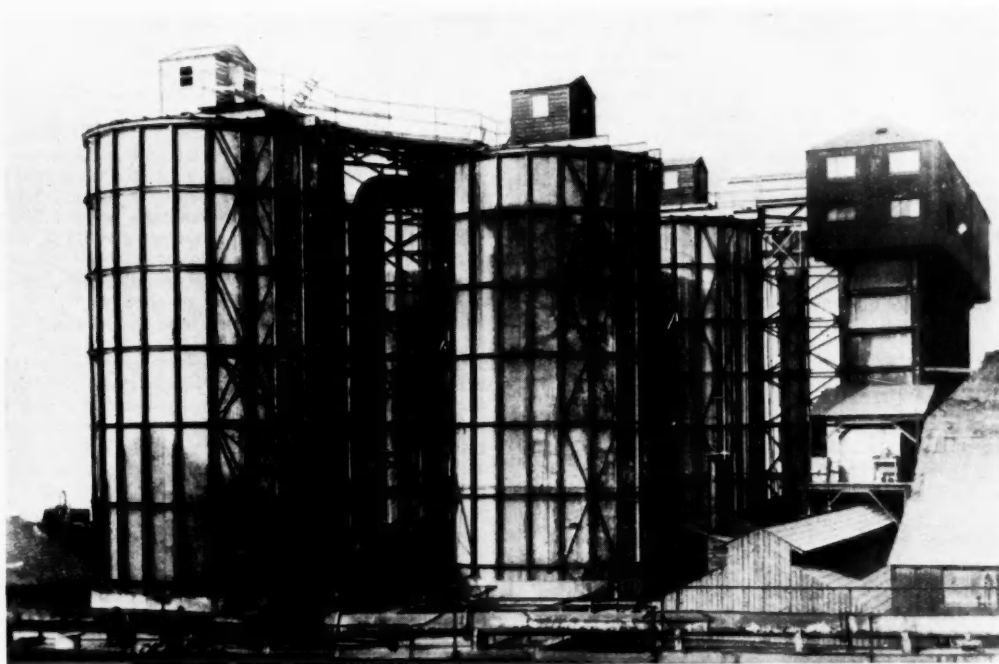
Phosphate and Superphosphate

The progressive development of the phosphate industry during the last twelve years is characterised by three different processes, namely: (1) The enrichment of inferior phosphate by flotation methods; (2) the manufacture of triple superphosphate, and (3) the fixation of ammonia by superphosphate. In the United States of America there is a tendency to regard the ammoniating of superphosphate as the most important of these developments. In 1929, 30,000 tons of nitrogen were consumed for this purpose, and the estimated consumption for 1930 is 40,000-50,000 tons. This is equivalent to 1.3-1.6 million tons of superphosphate, assuming the product to have a nitrogen content of 3 per cent.

Earlier attempts to produce ammoniated superphosphate were abandoned, owing to the reconversion of the water-soluble phosphoric acid, but later experiments have definitely established the fact that this retrogression does not affect the efficacy of the fertiliser to any appreciable extent. The fixation of ammonia by mono-calcium phosphate and phosphoric acid is accompanied by the evolution of heat, which is sufficient to evaporate a proportion of the water contained in the superphosphate. The product is, therefore, dry, and in excellent condition for distribution. Moreover, the material does not attack the sacks.



MONT CENIS SYNTHETIC AMMONIA FACTORY: THE COMPRESSOR HOUSE.



THE GAILLARD-PARRISH MODERN EXPOSED TYPE LIQUID PHASE SULPHURIC ACID PLANT.

If over 40,000-50,000 tons of nitrogen in the form of an aqueous solution of ammonia can be used by the superphosphate manufacturers in America, it is certain that co-operation between the superphosphate industry of this country and the gas undertakings and coke oven works, would be well worth while. Gas liquor can be converted inexpensively to an ammonium bi-carbonate solution, or to ammonia liquor, sufficiently pure for the ammoniation of calcium superphosphate without much expense, and it would appear that collaboration between the two industries would prove mutually advantages. The cloud which pervaded the superphosphate industry last year has not lifted: indeed, matters have become rather worse, than better.

Whatever governmental support is given to agriculture (and such support is being anxiously awaited), it is certain that calcium superphosphate manufacturers will benefit, as indeed they must profit by the devaluation of the pound sterling, which must have partially, if not completely, checked the importation of foreign superphosphate into this country. The future largely depends upon the wisdom of the National Government, and on international sanity. Both the standstill agreement, and the Hoover moratorium for war debts and reparations, must be continued. France must adopt a rational attitude, and cannot be allowed to stand in the way of peace and prosperity, which are so essential to the recovery of world trade, and incidentally, to the rehabilitation of the heavy chemical industry.

The British Synthetic Fertiliser Industry

Progress at Home and Overseas

The slowly accumulating data obtained by research, whether in the manufacture or in the use of fertilisers, grows in importance as time goes on. This article reviews the progress which has been made during the past year, both at Home and Overseas.

THE trade difficulties of the past twelve months have naturally been reflected in the synthetic fertiliser industry. Notwithstanding these, however, great progress has been made both in production methods and research and the close of 1931 finds the industry more firmly entrenched than ever and full of hope for the future. That there are strong grounds for these hopes may be proved by the fact that already the forward bookings in the home market for 1931-1932 exceed the total sales for the whole of the past season.

Ammonium Sulphate

The break-down in the European nitrogen agreement, the general agricultural depression and the price reductions consequent on these events, had the effect of forcing many producers of bye-product ammonium sulphate to seek alternative methods of liquor disposal. The depreciation in the pound sterling has caused a slight re-adjustment in prices and this factor, in conjunction with the small improvement recently shown in the heavy industries, has made it possible for some of the larger coke oven producers once more to enter the market.

Generally speaking, the home trade for sulphate showed a slight decrease but the export market was well maintained. During the year, out of a total production of 545,972 tons, 456,467 tons were exported. Shipments to China and Japan, although, as always, very heavy, showed decreases, but Java, Ceylon, the Canary Islands, Spain, Portugal and some other countries all took greater supplies than in 1929-1930.

In a similar review last year, reference was made to research then being conducted with the object of improving the physical condition of ammonium sulphate and reducing the tendency of this material to cake during storage or shipment overseas. The work continues and already the product has been improved both in colour and size of crystal. It is hoped that in 1932 an even whiter sulphate in still larger crystals will be put on the market.

Nitro-Chalk

Large increases have taken place in the sales of this product at home and overseas and it is everywhere being recognised as a great improvement on all other fertilisers primarily intended for top-dressing. Its British origin has greatly strengthened its position in this country, whilst abroad its lime content and lasting effect in the soil have resulted in notable increases in consumption.

During the past year, much attention has been given to methods of improving the physical conditions of Nitro-Chalk. As a result, it has been found possible to produce a more uniform granule and, at the same time, to improve the storage properties. For greater convenience in handling, Nitro-Chalk is now packed in 1 cwt. moisture-proof bags.

Synthetic Nitrate of Soda

The large-scale manufacture of this product from synthetic nitric acid and soda ash, began at the Billingham factory of Synthetic Ammonia and Nitrates Ltd., during the year. It is made in crystalline form and is guaranteed 98.5 per cent.

pure. For the benefit of those farmers who, notwithstanding the lower price and other advantages of Nitro-Chalk, prefer nitrate of soda, a limited supply of this material will be available in the home market in 1932.

Concentrated Fertilisers

These fertilisers were described in some detail last year and it is, therefore, only necessary to say that their popularity continues, based, doubtless, on the economy effected in transport and handling on the farm. Demonstrations and trials in many parts of the country have indicated their suitability for most soils and climates.

In the export market, the most popular member of the concentrated fertiliser group is the Type B. ammonium phosphate (18 per cent. N., 18 per cent. P_2O_5). This is sold overseas under the name of Niciphos and is in demand for manuring rubber, tea, coffee, bananas, cacao and other tropical and semi-tropical crops.

Agricultural Research

At Jealott's Hill Research Station, the synthetic fertiliser industry continues to expand its knowledge by agricultural research. The interest taken in the Station by research workers and farmers continues. More than 1,000 visitors made the tour of the Farm and Laboratories during the past year. Of these 150 came from abroad.

Much new work was undertaken in 1930 and many laborious researches into obscure problems were continued. As an example of the tasks undertaken by Sir Frederick Keeble and his staff, it may be of interest to mention that recently a mature banana plant, complete with the bunch roots, and surrounding soil, was shipped from the Canary Islands for analysis and examination. Grassland continues to form an important part of the research programme and experiments on different types of pasture are always in progress. When to apply fertilisers to cereal crops in order to gain the most profitable increase in yield, is also a subject on which more light is being sought. It is already apparent that the problem of lodging may be attacked along these lines and that by delaying the nitrogenous top-dressing until the period of most vigorous growth (about mid-May) the grain may be given full increase without a corresponding growth in the straw. Rate of application, width of spacing and variety or strain of plant are other factors whose effect and interplay are being studied in the field.

Investigations into the problems of fruit manuring form an important branch of the work at Jealott's Hill. The apple orchard, planted according to the latest experimental technique, will, in years to come, provide data on the fundamental principles of apple manuring. Clonal or pure line vegetatively propagated trees, form the basis of the Jealott's Hill experiment. The slowly accumulating data obtained by research, whether in the manufacture or in the use of fertilisers, grows in importance as time goes on. For the moment, however, prospects for the employment of this new knowledge depend on a remunerative level of prices being obtainable by the primary producer.

Association of British Chemical Manufacturers

A Survey of its Activities and those of its Related Bodies

The activities of the Association of British Chemical Manufacturers, which is the most central and influential organisation in the industry, are described in this article. Additional notes are added concerning its affiliated Associations.

THE past year has been one of considerable activity for the Association of British Chemical Manufacturers. The main problems of the past few months have arisen from the economic crisis which has directed special attention to the protection of our home industries by means of tariffs. A detailed investigation of the imports of chemical products has been made with a view to ascertaining the approximate amount of production and employment that might be expected to accrue to this country from the imposition of tariffs on chemicals. The figures for 1929, the last year for which the full data were available when the investigation was made, showed that after making all due deductions for products which could not be obtained or made here and for imports which were at present coming in, notwithstanding the protection afforded by the Key Industry Duty on fine chemicals and the Dyestuffs Act, there was a balance of some £9,000,000 worth of goods which could, without any doubt, be made in this country. The adoption of a scientific system of tariffs on these products should lead, if the imports were replaced by home manufacture, to a direct increase in employment of the order of 20,000 to 25,000 men. Apart from the indirect effects of this employment on the national prosperity, the reductions in price which ought to result from the increased volume of production should facilitate an improvement in the export trade, and in the activity of the industries consuming chemicals. On these figures, it was clear that there was a case for the consideration of tariffs as a possible means of assisting the chemical industry without hardship to the consumer. As a corollary to this review, the Association has given detailed consideration to the grades of duty which should be applied to chemical products in the event of a general emergency tariff being adopted by His Majesty's Government. The proposals of the Association will be submitted to the Government as soon as circumstances demand.

National Economic Crisis

With the return of the National Government to power, with its mandate to redress the adverse balance of trade, the foreign suppliers became apprehensive that a tariff system was imminent. They have, therefore, been taking active steps to forestall this contingency by dumping large quantities of goods in this country. The chemical industry has not been immune from such attacks and details of a number of clear-cut cases, which could be substantiated by accurate import figures, were brought to the notice of the Government in order that they might appreciate the danger of any delay in protecting the British manufacturer. Such dumping not only intensifies our unemployment problem, but, by increasing our adverse trade balance, already exaggerated by our departure from the gold standard, imperils the position of the £ sterling. The National Government acted promptly, and by means of the Abnormal Importations (Customs Duties) Act, conferred the necessary powers on the President of the Board of Trade to impose duties in all cases in which he was satisfied that the imports were made with the intention of forestalling a tariff policy. While the President was given a free hand in his interpretation of "abnormal," he has, as yet, used his powers only in a very restricted field, and in relation to only a limited number of sections of industry. At the moment of writing, none of the orders deals with products of interest to the chemical industry. There is some doubt as to whether the President will endeavour, under the Act, to check the imports of an abnormal nature which have been coming in for some considerable time, to the detriment of British manufacturers.

Our departure from the gold standard has raised a number of other problems with which the Association has had to deal. In the first place, difficulties arose in regard to international contracts owing to the attempts of some foreign traders to repudiate contracts made on a sterling basis. The Association advised its members that the validity of contracts was not affected by changes in currency, and urged that nothing

should be done to prejudice this position. At the same time, it sought the support of the corresponding organisations in the more important chemical manufacturing countries, pointing out that all contracts are being honoured on the British side, even when they involve a loss, and asking for their co-operation and assistance in securing the similar observance of contracts by their nationals. It is gratifying to report that those who have replied have adopted a most sympathetic attitude. The Association is arranging to keep a list of firms who default on their contracts for such action and publicity as may appear desirable at a later date.

The depreciation of our currency has given a powerful impetus to the policy of Buying British, of which the Association has for long been a strong advocate. For some considerable time, the Association has urged the adoption of the policy through various trade organisations which are important purchasers of chemicals, and has obtained a considerable measure of support. The national "Buy British" campaign which has now been inaugurated will be of the greatest value to our industries, coming as it does at a time when our depreciated currency is proving a compelling argument for those British buyers whose pockets have, in the past, unfortunately overruled their patriotism. The help which has been given to the home and export trade by our departure from the gold standard can, however, be only of a temporary nature and will, in any case, disappear as other countries follow our example. Furthermore, the increase in the cost of the food and raw materials, which must be imported, will slowly but surely reduce the advantage we now hold. It is, therefore, of the utmost importance that this lag should be as long as possible and, with this end in view, the Association has recommended to its members that prices should not be increased unless and until fully justified by prime costs. There is every reason to believe that British manufacturers are playing the game and carrying out this policy, and it is up to consumers to see that traders and middlemen do not exploit the position. As regards the export trade, every effort must be made to regain our lost position. British industry as a whole must be completely overhauled in all its aspects. The utmost efficiency must be secured and every possible wastage of effort must be eliminated. To this end, attention is being given to co-operative effort as regards buying of raw materials, manufacture and selling of final products. Co-operative selling in overseas markets requires intensive study as all the British missions of the last few years have drawn attention to the lamentable weakness of our selling arrangements. In short, the Association is giving consideration to every possible means likely to improve the home and export trade in chemicals and to ensure that all chemicals required by British users are made by British industry.

Inter-Imperial Industrial Co-operation

The present crisis has brought to the fore the old question of inter-imperial industrial co-operation. This is a policy with which the Association is in strong sympathy and on which it has already expressed its views to the Government. The basis of the policy is the recognition of the right of every part of the Empire to develop its resources, in order to provide employment for its increasing population. Such development has not always been on sound economic lines in the past, and there has been a tendency to start new manufactures which, for one reason or another, could never be in a position to withstand world competition and to protect them by high tariffs, with serious repercussive effects on the well-being of that particular community as a whole. The suggested form of co-operation is that British industry should collaborate in the formulation and execution in other parts of the Empire of such schemes as are commercially sound, by providing financial and technical resources; in return for this, special preferences would be given to British products which cannot be made economically in that particular part of the Empire. There is plenty of scope for such co-operation

in the chemical field as can be seen from the importation from foreign sources of over £20,000,000 worth of chemicals, most of which could be made inside the Empire. This type of inter-imperial industrial co-operation is already being carried out in certain directions, but its energetic adoption as an imperial policy could not fail to benefit the Empire as a whole. It is hoped that the next Imperial Conference will see this policy accepted.

Safety in Industry

The Association has continued its safety activities in the form of its Model Safety Rules, its Safety Circulars, and its Quarterly Safety Summary. The study of safety is regarded by the Association as on the same plane as medical science. The Association has, therefore, made arrangements by which its work on safety will be at the disposal of firms or organisations outside the Association which are willing to co-operate.

In order to further the cause of safety, the Association organised what was the first Chemical Session in connection with the National Safety First Week of the National "Safety First" Association at Leeds in May. The Chairman of Council presided, and the general manager delivered a paper dealing with the principles to be observed in the cleaning and repair of plant and vessels containing dangerous material. This was followed by a successful discussion in which valuable contributions were made by representatives of the tar distilling, dyestuffs, artificial silk, dry cleaning, and synthetic high pressure industries, and of the Factory Department of the Home Office, on problems arising in the application of the principles enunciated in the paper. Wide publicity was afforded to this discussion by the Technical press so that the mass of useful information contained in the various contributions has been brought to the notice of the largest possible circle of chemical technologists.

In the cleaning of vessels where there is a danger of gassing, the use of white mice has been recommended by the Home Office as a suitable test of safety. The reliability of this method, depending as it does on the assumption that the white mouse is at least as sensitive as man for the various toxic agents encountered in industry, was queried on physiological grounds, and the subject is now being investigated by the Association and the Home Office in conjunction with the Chemical Defence Research Department, which has a unique experience of the action of poisonous gases on various types of animals.

The Home Office has recently appointed a Departmental Committee to consider whether poisoning by carbon monoxide and hydrogen sulphide and their sequelae should be scheduled as industrial diseases under the Workmen's Compensation Act. The Works Technical Committee of the Association has made a detailed investigation of this problem on behalf of the Association of British Chemical Manufacturers, the Association of Tar Distillers, and the Chemical and Allied Employers' Federation, and has come to the definite conclusion that there is no evidence which would support the proposal that poisoning by these gases should be scheduled. A mass of data in support of this view has been secured for presentation to the Home Office Committee.

Standardisation

The standardisation movement initiated by the Association in respect of chemistry had been brought to a successful conclusion, and has resulted in the expansion of the British Engineering Standards Association to form a British Standards Institution covering the whole of industry. The new organisation will be controlled by a Grand Council and will consist of four co-equal and autonomous divisions, each with its own Council, and dealing respectively with engineering, chemistry, building and textiles. The constitution of the chemical division has been settled, and a provisional chemical council appointed over which the chairman of the A.B.C.M. Council, Dr. E. F. Armstrong, presides. The new council is now reviewing the whole chemical field and collecting and collating the work of standardisation which has already been done, with a view to formulating a programme of work to meet the needs for standardisation in the wide field of industrial activity represented by the chemical division.

Patents

The Board of Trade Patents Committee to which the Association, in conjunction with other interested chemical organ-

isations, submitted a memorandum at the end of 1929, has now issued its report. Most of the proposals advocated by the chemical interests were adopted, the main exception being in the case of medical patents where the Board of Trade felt that the scheme of dedicated patents was adverse to British research workers and industry. The objects to be achieved by this scheme have even now been imperfectly understood. The difficulties regarding medical patents are largely psychological and they have not been removed by the recent resolution passed by the British Medical Association defining its ethical attitude to such matters. Until the whole position of British medical patents is clarified, which it must be in the national interests, the British research worker and industry will continue to be heavily handicapped against the foreigner in the development of new medical remedies. An important advance for which the Association has made repeated representations since 1922 has now been made by the Medical Research Council. This Council has organised a system for the proper clinical testing of new products by means of a specially constituted Committee, which will conduct all the arrangements with the clinicians to whom the work is to be entrusted. These arrangements will remove a handicap from which this country has long suffered, as compared with our Continental competitors.

The Dyestuffs Act

With the continuation of the Dyestuffs Act under the Expiring Laws (Continuance) Act, 1930, the dye makers at once renewed their offer to the users that they were prepared to meet the fair foreign price for an equivalent product, failing which they would not oppose the issue of a licence. This offer was accepted by the Dyestuffs Advisory Licensing Committee, and is now the basis on which the price cases have been considered during this year. At the same time, the makers made a further proposal, namely, that applications for licences should be granted for new colours in reasonable quantities immediately they were received, so that the possibility of any delay to colour users in obtaining novelties for their trade might, in this way, be obviated. These concessions have removed the last trace of economic hardship, of which the users have complained in the past. The makers are also endeavouring, by co-operative means, to reduce the imports of those dyes which are still being imported in substantial quantities, and in this connection, such steps as were possible have been taken to reduce overlapping and duplication of effort. The Colour Users' Association having expressed their preference for the continuation of the Act rather than a system of tariffs, the Act has been extended for another year under the Expiring Laws (Continuance) Act, 1931.

Transport of Chemicals

The Traffic Committee of the Association continues to do valuable work in connection with the transport of chemicals by land and sea, and has been able to secure considerable ameliorations in the rates and conditions of carriage. The Association is represented by its General Manager on the Board of Trade Committee which is revising the conditions for the transport of dangerous goods by sea, with a view to the submission of a memorandum as the basis for an international convention on this subject. This work is of the greatest importance to the chemical industry, as much trade has been lost in the past owing to the more stringent conditions as regards packages and stowage imposed by the Board of Trade Regulations, and their more rigid adoption by our shippers. The promulgation of international rules on this subject will put all competitors on the same basis, and so remove one serious handicap to our trade. The Association has been engaged in working out a co-operative scheme for the insurance of private wagons which, when completed, will result in considerable economies to the membership as a whole.

The above represents only a few of the major problems with which the Association has dealt during the past year, and is far from being a comprehensive survey of its activities. One other important service which it has done was to publish an alphabetical Index to the Acts of Parliament and Statutory Rules and Orders affecting the chemical industry, with the idea that this should form a ready guide and reference book to any manufacturer who desires to ascertain quickly what his statutory obligations are in any particular

direction. This novel publication has been much appreciated, and arrangements have been made to keep it up to date by periodical addenda. It can be purchased by non-members as well as members.

At the annual general meeting in July the officers of the Association were re-elected for a second year as follows:—President, Mr. C. A. Hill; vice-president, Dr. G. C. Clayton; chairman, Dr. E. F. Armstrong; vice-chairman, Mr. D. Lloyd Howard; hon. treasurer, Mr. E. V. Evans.

The Association of Tar Distillers

The Association of Tar Distillers, through the National Creosote Committee, has continued to devote much attention to the problem of finding new outlets for creosote. In this connection it has given its cordial support to the British Wood Preserving Association which, though only in its second year, is already doing most valuable work of national importance in spreading knowledge in regard to wood preservation. The British Wood Preserving Association has not only arranged lectures and discussions of subjects of interest and published a volume of proceedings, but has also exhibited at the more important agricultural shows throughout the country. The exhibit, which affords convincing proof of the value of timber preservation not only for specialised purposes but also for the numerous uses which touch the public at large, has been warmly praised in all quarters and has been awarded several medals. The Association has also undertaken the production of an educational film dealing with the whole life history of timber, which will be a most valuable means of spreading knowledge.

One of the main objections to the use of creosote for the treatment of timber arises from the fact that, so far, no suitable means has been found of painting wood which has been impregnated, as distinct from merely brushed over, with creosote. Many attempts have been made to solve this problem. The National Creosote Committee has been able to secure an option on a new method which holds out reasonable promise of success, and is now conducting long period tests to observe the behaviour under various conditions of weather and use of creosoted wood painted by the new process.

The National Creosote Committee has kept closely in touch with, and has given encouragement and assistance to, the work which is going on in many quarters on the use of creosote or tar oils in engines, both of the Diesel and ordinary internal combustion type, and has acted as a co-ordinator and clearing-house of information.

The Association has also given careful consideration to the national economic situation and has drawn up a memorandum to indicate its views as to the tariffs which should be applied to coal tar products, in the event of the National Government deciding to impose a general emergency tariff of a protective nature. Its views will be submitted to the Government at the appropriate moment.

The rapid corrosion of tar stills is a problem which has become more acute of recent years owing to some factor which is thought to be connected with changes in the nature of the tar arising from the introduction of new methods of carbonisation. The Association of Tar Distillers has therefore appointed a Tar Stills Corrosion Committee to investigate the whole problem, on which the resistant Metals Committee of the Association of British Chemical Manufacturers and the British Chemical Plant Manufacturers' Association will be represented. After a preliminary review it was realised that the subject was of such complexity that it would be advisable, as a first step, to collect the whole of the relevant data from the tar distilling industry of the country. A suitable questionnaire was framed and the collection of the desired information is now nearing completion. The Tar Stills Corrosion Committee has been very fortunate in securing the co-operation of Mr. W. A. Damon, the chief alkali inspector, whose last report contained a valuable appendix descriptive of the research work which was being done in his department on this problem.

The Association has played an important part in the formation of the new British Standards Institution and is represented on the Council of the Chemical Division of the Institution by Mr. H. W. James, of South Metropolitan Gas Co.

A new directory has been issued in the last few months setting forth particulars regarding the offices and works of

the members, and is available to anyone interested in tar products on application to the Secretary.

At the annual general meeting in March, Mr. R. Alsop, of Consett Iron Co., Ltd., was elected President in succession to Mr. F. C. T. Tudsbury, of Scottish Tar Distillers, Ltd., while Major A. G. Saunders, of Burt, Boulton and Haywood, Ltd., was re-elected hon. treasurer. The new vice-president is Captain Ward-Jones, of the Rothervale Collieries Branch of the United Steel Companies, Ltd.

British Chemical Plant Manufacturers' Association

The most noteworthy activity of the British Chemical Plant Manufacturers' Association during the year under review has been the organisation of the very successful exhibition of chemical plant and apparatus, which was held at the Central Hall, Westminster, in July, during the jubilee week of the Society of Chemical Industry. The exhibition was well supported by British chemical plant makers and all the available space was booked. The exhibits covered the whole range of chemical plant and comprised a large number of novel designs and constructional materials which were on show for the first time. The Association was most fortunate in securing the close co-operation of the Society of Chemical Industry in all its publicity arrangements, and of the Chemical Engineering Group which organised a most interesting section to demonstrate the work of the Research Associations operating under the aegis of the Department of Scientific and Industrial Research. There was a gratifying attendance of people directly interested from all over the country and this enabled the chemical plant manufacturers to show the present and potential users of their goods that, in design and construction, this country has nothing to learn from the foreigner.

The exhibition has received such wide and detailed notice in the technical press that further description is unnecessary. The exhibitors have been unanimous in expressing their satisfaction with the results of this venture, which has earned the outspoken approbation of the organisers of the German Achema. In the new year consideration will be given to the question of the date and general arrangements for the next exhibition. At the moment, the general feeling seems to be in favour of an exhibition every third year, so arranged that it will not clash with the similar exhibitions which the French and Germans propose to hold on a three-yearly basis.

Practically every British mission which has gone abroad in the last few years has returned with the criticism that this country is losing its trade because of its weak selling arrangements. The policy of co-operative selling has been repeatedly advocated as the best means of rectifying this defect. The Association has therefore instituted a careful investigation into the whole subject with a view to determining precisely whether and how far co-operative methods are advisable in the marketing of chemical plant. The problem is a complex one, but it is hoped that useful results will emerge. Attention is also being given to the possibilities of developing trade in certain markets.

Japanese Ammonium Sulphate Position

It is reported that stocks of foreign ammonium sulphate in Japan on October 20, amounted to 43,000 tons. Imported supplies were being offered at 59 yen per ton c.i.f. ports, while the quotation of a domestic producer, namely Nihon Chisso K.K., was 61 yen per ton wholesale on cars. The Mitsui company will control practically 50 per cent. of domestic production in Japan by the end of 1931. The Miike plant, one of the Mitsui operations, has a production of 30,000 tons, but it is anticipated that this will be increased to 100,000 tons during the next year. Other Mitsui enterprises are Electro-Chemical Co., 70,000 tons; Nihon Chisso and Chosen Chisso, 100,000; Mitsui Mining, Kamaishi Mining and Nihon Steel Works, 8,000 tons.

Sulphur and Pyrites in Sweden

IMPORTS, during the June quarter of 1931, of sulphur and pyrites, which are utilised largely by pulp mills, were 9,513 metric tons and 31,649 metric tons respectively. During the same quarter of 1930, 16,957 tons of sulphur and 49,116 tons of pyrites were imported. Consumers generally fill their stocks during the summer months when imports may be directed by inland canals and other waterways into the interior.

Standardisation in Chemical Industry

Progress made by the British Standards Institution

In order to provide for the widening of its scope and activity the constitution of the British Engineering Standards Association has recently been enlarged, a Supplemental Charter being granted under which the present Association is now recognised as the national organisation for all industrial standardisation with the title changed to British Standards Institution.

THE past year has been one in which developments of far reaching importance have taken place in the growth of standardisation in the chemical industry. The desirability of instituting an executive standardising body responsible for furthering and controlling work in the preparation of standards for the assistance of and benefit to the chemical industry was very emphatically expressed at the fully representative conference which was convened in June, 1930, under the aegis of the Association of British Chemical Manufacturers.

Co-incident with this urge for standardisation from the chemical industry, came the unanimous recommendation of the Imperial Conference that there should be one national standardising organisation in all the various great branches of the Empire who should have in the foreground of their programme the co-ordination of specifications within the Empire. The British Engineering Standards Association have endeavoured to meet this new position and the increasing demand for standardisation, and with the most cordial co-operation of the Government Departments and all the principal technical and trade organisations in the chemical industry a Chemical Section of the Association has been formed. The existing Engineering Committees have therefore been grouped into one Division controlled by an Engineering Divisional Council and similar Divisional Councils have been formed for the chemical, building and textile industries.

The Chemical Divisional Council

The Chemical Divisional Council is constituted as follows: Mr. F. G. Edmed, O.B.E. (Admiralty); Dr. D. R. Pye (Air Ministry); Mr. L. M. Nash (Board of Trade); Mr. G. H. Perry, O.B.E. (War Office); Dr. E. F. Armstrong (Chairman), Mr. F. W. Lewis, Colonel E. Briggs, Dr. R. E. Slade, and Mr. J. Davidson Pratt, O.B.E. (Association of British Chemical Manufacturers); Mr. H. W. James (Association of Tar Distillers); Mr. V. Blagden (British Chemical & Dyestuffs Traders' Association); Dr. H. J. Bush (British Chemical Plant Manufacturers Association); Dr. J. W. Mellor (Ceramic Society); Professor W. A. Bone (Chemical Society); Professor G. T. Morgan, O.B.E. (Chemical Research Laboratory (D.S.I.R.)); Mr. W. Rintoul, O.B.E. (Federal Council for Chemistry); Mr. T. Macara (Food Manufacturers Federation); Mr. G. Marchand (Glass Manufacturers Federation); Dr. J. J. Fox, O.B.E. (Government Laboratory); Mr. S. A. Brazier (India Rubber Manufacturers Association); Mr. W. J. A. Butterfield (Institute of Chemistry); Dr. F. A. Freeth, O.B.E. (Institution of Fuel); Dr. A. E. Dunstan (Institution of Petroleum Technologists); Mr. A. B. Craven (International Society of Leather Trades Chemists (British Section)); Mr. A. H. Berlandina (London Oil and Tallow Trades Association); Mr. W. G. Adam (National Benzole Association); Sir C. A. Mander, B.T. (National Federation of Associated Paint, Colour & Varnish Manufacturers of the United Kingdom); Mr. H. N. Linstead (Pharmaceutical Society); Mr. W. A. S. Calder (Society of Chemical Industry); Mr. J. Blair (Society of Dyers & Colourists); Mr. E. R. Bolton (Society of Public Analysts); Sir Alexander Walker, Lord Leverhulme, Lord Trent and Sir John Cadman (Co-opted members).

The administrative and executive control of the British Standards Institution is vested in a General Council on which each Divisional Council is represented by nine members and at the first meeting of the Chemical Divisional Council the following members were elected as the representatives of the chemical industry on the General Council: Dr. E. F. Armstrong, Mr. W. B. Craven, Dr. A. E. Dunstan, Sir Herbert Jackson, Mr. H. W. James, Lord Leverhulme, Sir Charles Mander, Sir David Milne Watson, Mr. W. Rintoul.

It is most gratifying to know that the services of Dr. E. Frankland Armstrong have been secured as Chairman of the Chemical Divisional Council and under his initiative and direction the success of the development of this important and

long overdue adjunct to the chemical industry can be confidently anticipated.

Work Already Accomplished

Preliminary work has involved a complete survey of all chemical specifications in existence both those issued by industrial corporations and by institutions. The urgent need for many of these to be issued as British Standards has been already made manifest and steps are being taken in an endeavour to expedite their publication as such. This re-organisation has in no way diminished the activity of the work which was already in hand. Several important specifications of direct and immediate interest to the Chemical Division have been issued during the past year amongst which the following might be mentioned:

1. *Test Sieves.* After a very careful investigation of the existing position by a thoroughly competent committee, the need for evolving a satisfactory system of test sieves was revealed, and this has now been met by British Standard Specification No. 410—Test Sieves. It is very encouraging to be able to report that the Institution of Mining and Metallurgy—the pioneers in sieve standardisation and who co-operated in the present work—have recommended their members to use the British Standard Sieves wherever possible.

2. *Gas Cylinders and Fittings.* Three British Standard Specifications, Nos. 399, 400 and 401, covering Gas Cylinders for Permanent and Liquefiable Gases have been issued. The requirements of these specifications have been based on the recommendations of the D.S.I.R. Committee as embodied in their First and Fourth Report. In addition, a British Standard Specification for Valve Fittings and a standard colour scheme for the Identification of Gas Cylinders have also been issued in Specification No. 341 and 349 respectively.

3. *Sampling and Analysis of Coal.* A specification dealing with the Sampling and Analysis of Coal for Inland Purposes has been issued as B. S. Specification No. 420, which like B. S. Specification No. 404, Coal for Export, is based on the principles of sampling enunciated by Dr. Grummel and Dr. Dunningham in B. S. Report No. 403.

4. *Non-Ferrous Metals.* The following further British Standard Specifications for Non-Ferrous Metals will doubtless be of interest to those chemical engineers associated in any way with metallurgy: B.S.S. No. 407. Phosphor Bronze Sheets and Strips. B.S.S. No. 404. Naval Brass Plates, Sheets and Strips. B.S.S. No. 414. Wrought Light Aluminium Sheets and Strips (Y Alloy).

Future Programme

Features of interest amongst the items of work commenced during the year, the progress of which is proceeding satisfactorily, include the preparation of draft specifications for (a) the identification of chemical pipe lines; (b) the standardisation of containers, including glass carboys, metal drums, etc., and tank wagons; (c) carbide of calcium; (d) welded containers for permanent and liquefiable gases; (e) the size, position and shape of access openings in chemical plant; (f) chemicals and chemical plant for electroplating.

It will therefore be seen that, despite the changes which have been taking place, the work of standardisation, of which there is at the present time more than ever a pressing need, has therefore been proceeding most satisfactorily.

Increase in Indian Salt Production

The year has recorded a marked increase in the total quantity of salt imported into Bengal and prices have been steadily on the decline. The consumption of salt imported from the United Kingdom and Aden has dropped considerably, while that from Hamburg, Port Said and Spain shows a big increase. A point of note is that there is a further increase in the consumption of both Bombay and Karachi salt and the latter secures a steady market.

The Dyestuffs Situation

The Work of the Colour Users' Association

In this article the dyestuffs situation is discussed from the point of view of the colour user. The work of the Association during the past year has been mainly concerned with the Dyestuffs Advisory Licensing Committee and the extension of the Dyestuffs (Import Regulation) Act.

DURING the past twelve months the Association has been chiefly engaged in examining the procedure of the Dyestuffs Advisory Licensing Committee with a view to making effective the concessions offered by the manufacturers previous to the extension of the Act last year—namely that the pre-war factor ratio should be abolished and that licenses should only be refused in those cases where British makers were prepared to supply equivalent products at similar prices to those quoted by foreign manufacturers, so long as those prices were not "dumping" prices.

These proposals have been adopted by the Licensing Committee, and it was arranged that in the case of applications on price grounds licenses to import should be issued if British makers did not indicate within 48 hours of being notified of the foreign quotation their willingness to supply at the same price. If an objection was lodged to the quotation as being in the nature of a "dumping" quotation, it was agreed that a licence should be granted to the applicant to import a sufficient quantity of the dyestuff in question to enable him to carry on while the matter was being investigated.

Unfortunately, experience has proved that in practice the offer of the British makers to meet foreign prices has been nullified by the fact that foreign manufacturers refuse to quote on these terms. After all, it is hardly reasonable to expect a foreign producer to submit his lowest prices, with the knowledge that his quotation will immediately be shown to competitors who will have the option of refusing or accepting the business at the same price. Unless the ability of the British consumer to obtain competitive foreign quotations can be established, it would appear that the result of the existing procedure will be to debar users from access to competitive prices for nearly 80 per cent. of British requirements. Negotiations are at present in progress with a view to rectifying this state of affairs which has prevented the British producers' offer—the sincerity of which is not disputed—from being effective. The Council of the Colour Users' Association have urged that in future foreign quotations should only be disclosed to neutral members of the Licensing Committee and that applications for licences on price grounds should be dealt with on a strictly competitive basis as between foreign and British makers without the British makers having an option to meet the foreign price. The results of the existing method is to give the British makers a monopoly, which no doubt was not intended when the proposal was made.

Extension of the Dyestuffs Act

When the last yearly review was published in THE CHEMICAL AGE the future of the Dyestuffs Act was still in doubt and the subject of keen controversy. It will be remembered that the expiration of the Act was debated in the House of Commons on December 4, 1930, when an amendment to include it in the Expiring Laws Continuance Bill and so prolong it for five years, was defeated. On December 15 an amendment was passed in the House of Lords to include the Dyestuffs Act in the Expiring Laws Continuance Bill for one year pending an impartial investigation of the position. On December 17 the amended Bill was again considered by the House of Commons when the amendment to prolong the Dyestuffs Act for another year was hotly debated. The motion to reject the amendment put forward by the House of Lords was passed by a majority of six only, and on the following day (December 18) the Lords again insisted on their amendment, which was then accepted by the House of Commons.

At the Association's Annual Meeting in July last, the hope was expressed by the Chairman (Sir Henry Sutcliffe Smith) that the extra twelve months extension would have considerably strengthened the position of the British dyestuffs industry and enabled it to stand on its own merits independent of adventitious aid. The Council of the Association also discussed their position with regard to the Dyestuffs Act at their meeting on September 22 last and re-affirmed their opposition to any extension of the Act.

Since that date, however, the situation has been completely changed owing to recent political developments, and at the meeting of the Council of the Association held on November 6, 1931, the following resolution was unanimously passed:—

"The Colour Users' Association consider that any tariff on dyewares—one of their important raw materials—would be prejudicial to their industries and whilst not receding from their declarations regarding the Dyestuffs (Import Regulation) Act, they agree that in present circumstances a continuation of that Act for a further twelve months would be preferable."

The Dyestuffs Act was ultimately included in the Expiring Laws Bill, which was read for the third time in the House on December 3 last, and will now be extended for a further year.

Dyestuffs Industry Development Committee

A subject to which the Council has devoted a considerable amount of time during the past year is the consideration, in conjunction with the Dyestuffs Industry Development Committee, of the possibility of establishing a British Standardisation Mark for British dyestuffs. It is undeniable that there is a lack of uniformity in regard to standards of fastness but the whole question is very complex and many conflicting points of view have to be taken into consideration. A sub-committee of the Development Committee has been appointed to investigate the various problems and the consumers' views are being presented.

The Joint Technical Committee, composed of an equal number of representatives of makers and users, continues to do useful work in the consideration of new British colours put forward to replace foreign products which is of great assistance to the Dyestuffs Advisory Licensing Committee.

German Foreign Trade in Linseed Oil

IMPORTS of linseed oil into Germany declined from 19,149 metric tons in 1929 to 15,391 tons in 1930. A further drop in the import trade was evidenced for the first eight months of 1931 when the total importation of oil was 5,513 metric tons which compares with 11,532 metric tons for the same period of 1930. The Netherlands supplied almost the entire imports of linseed oil. Exports of linseed oil likewise dropped from 6,746 metric tons during 1929 to 4,213 tons for 1930. Shipments, however, for the first eight months advanced from 2,952 tons in 1930 to 4,274 tons in 1931. The principal countries of destination were Switzerland, France, Austria and Great Britain.

Sulphur in South Africa

IN 1930, imports of sulphur were 64,186 short tons valued at £127,000 compared with 44,397 tons valued at £87,000 in 1929. The 1930 imports comprise 26,337 tons of crude sulphur and 37,849 tons of flowers of sulphur. United States is the chief source of supply, with small quantities coming from the United Kingdom, Belgium and Italy. The bulk of the crude sulphur imported is used in making sulphuric acid intended for superphosphates and for battery purposes. Considerable ground sulphur is used as sheep dip.

U.S. Trade in Sodium Cyanide

THE United States exports of sodium cyanide during the first nine months of 1931 were 452 short tons valued at \$130,530 compared with 436 tons valued at \$125,788 for the preceding year. The United States imports of this commodity were 7,102 short tons valued at \$1,266,549 in 1930 compared with 10,935 valued at \$1,656,456 for the same period of 1929.

Production of Borax in Italy

PRODUCTION of borax in Italy during 1930 was 4,826 metric tons valued at £98,000, according to the annual report for 1930 by the Mining Corps in the Ministry of Guilds. Labourers employed in the borax industry numbered 447.

Chemical and Allied Societies : Their Work During the Year

The following specially contributed reports on the work of the principal chemical and allied societies indicate the range and value of their work and record many important developments during the past year.

British Association of Chemists

THE Association has completed another very successful year of work. The increase in membership of 172 is regarded by the Council as very satisfactory and is indeed in excess of that recorded for many years. Smooth working of all the Association's activities accounts for this advance in numerical strength. The depressed condition of industry makes it essential for chemists to be members of an organisation which is in a position to give them assistance and advice upon all questions relating to employment. It is in this direction particularly that the Association has been able to support its members. New outlets for the application of chemistry to industry have to be found. The Association has been fortunate in exploring new spheres of activity for chemists by placing before employers the advantages gained from the service of the chemical practitioner and the importance of scientific control. Allowing for the exceptionally difficult conditions, the results have been highly gratifying.

Unemployment has, of course, increased as in the case of other occupations, but the percentage among members of the Association still remains extremely low. The Unemployment Benefit Fund has been of the greatest assistance to members who have had to call upon it, and the successful operation of the fund remains one of the greatest advantages membership can confer. The occupation of a highly paid and responsible position no longer carries with certainty any assurance of permanence. Many chemists who at one time criticised the scheme as being one inapplicable to their individual cases now recognise it as an activity worthy of the support of the whole profession.

The financial position both as to the General and the Unemployment Benefit Funds is thoroughly sound. In the case of the General Fund a small balance remains in hand on the year's working; in the case of the Unemployment Benefit Fund, although £900 has been paid out in benefit as against £520 last year, a substantial sum of over £800 has been placed to reserve. The satisfactory position of the General Fund is, of course, largely due to the increase in membership and to the rigid economy practised in the administration of the Association's affairs.

The Legal Aid Department has functioned very satisfactorily. Only one member of the Association has been obliged to proceed to court on the question of insufficient notice. The case in question resulted in a decision in favour of the member. A large number of agreements have been examined, and advice has been given to members in this and other directions.

At the end of last year a Memorandum on the safeguarding of dyestuffs was addressed to H.M. Government to which very careful consideration was given by those to whom it was addressed. Since the question of the extension of safeguards generally is now occupying the attention of H.M. Government, the Association hopes to be able to render the Government any further service that may be necessary in this connection.

The salaries secured for members through the service of the Appointments Bureau continue to be well maintained. In consequence of the general depression the activities of the Bureau have not developed to the quite exceptional degree attained last year, but it remains true that the conditions of service secured by members through the Appointments Bureau have been in a large number of cases much more satisfactory than they might otherwise have been.

The Association desires particularly to draw the attention of students to the importance of membership. A student while still at the university will discover that the Association can give him much valuable information regarding the conditions of industry and allied matters which will help him to form a conclusion as to the best course to pursue on the completion of his training. The annual subscription for students is five shillings only, on payment of which the student becomes eligible for all the privileges of membership except unemploy-

ment benefit, towards which the student cannot subscribe.

During the year the Registration Committee has reviewed and revised all committee reports and other memoranda relating to the subject of registration. The Association's policy concerning this matter has thus been brought completely up to date, and it has thus prepared recommendations which represent in the opinion of the Council a workable scheme. These reports have not yet been published since the time is not ripe, but the Council hopes that this information will make it quite clear that the matter has not been shelved.

The Association's propaganda and publicity has been consistently maintained both by means of its official journal and in the technical and non-technical press. The Council takes this opportunity of thanking the editors of all journals and newspapers who have published reports of the Association's activities.

Chemical Society

The Chemical Society, founded in 1841, has as its object the general advancement of chemical science by the discussion and publication of new discoveries. During the year under review, twelve Ordinary Scientific Meetings have been held, at which 65 papers were read and discussed, and the Second Pedler Lecture entitled "Studies in Biological Oxidation" was delivered by Professor Dr. H. Wieland, of Munich. An important innovation has been introduced this session, namely, to devote two Ordinary Scientific Meetings to organised Discussions on special subjects. The first of these was held on December 17, 1931, when a Discussion on "The Critical Increment of Homogenous Reactions" was opened by Mr. C. N. Hinshelwood, F.R.S. It has been decided that these Discussions shall be published as a separate number of the Journal. The volume will be sent free to Fellows and will be on sale to the Public. To commemorate the great services rendered to Chemistry and to the Society by the late Professor W. H. Perkin, F.R.S., the Council will publish early in 1932 a special memorial number of the Journal dealing with his life and work. The Faraday Lecture entitled "Chemistry and the Quantum Theory" by Professor Dr. Niels Bohr will appear shortly and will be available in separate form.

The Annual Reports on the Progress of Chemistry for 1930 (issued in February, 1931) contained, in addition to the usual reports, special reports on Geochemistry, Radioactivity and Sub-Atomic Phenomena, and on Electrical Conductivity of Solutions. The volume for 1931 (to be issued next February) will contain a biennial report on Crystallography and special reports on Covalency and on Colloid Chemistry.

The number of original communications published in the Journal of the Chemical Society is considerably higher in 1931 than it was in 1930, and there is also an increase in the number of abstracts in pure chemistry published in British Chemical Abstracts "A" issued by the Bureau of Chemical Abstracts.

The next Annual General Meeting and Anniversary Dinner will be held in Glasgow on Friday, March 18, 1932, the former taking place at the University and the latter at the Grosvenor Restaurant. The Council is gratified to report that on the occasion of this visit, Imperial Chemical Industries, Ltd., have generously invited those attending the meeting to visit their factory at Ardeer.

Application for research Grants amounting to £1,175 have been received during the year and sums amounting to £641 have been granted. The sum for which application is made each year far exceeds that available for distribution and applications from Fellows receive prior consideration.

The use of the Library steadily increases and the number of volumes, apart from pamphlets, is now 35,700. The Library is open daily from 10 a.m. to 9 p.m. (except on Saturday, when the closing hour is 5 p.m.) and is open to Fellows and to members of the following organisations: Association of British Chemical Manufacturers, Biochemical Society, Faraday Society, Institute of Brewing, Institute of Chemistry,

Society of Chemical Industry, Society of Dyers and Colourists, Society of Public Analysts.

Faraday Society

THE Faraday Society has this year only held one General Discussion, owing to the heavy programme of scientific meetings during the Autumn arising out of the Faraday, British Association and Clerk Maxwell Celebrations. The Liverpool meeting, when the General Discussion on "Photo-Chemical Processes" took place, was, however, extremely successful; many distinguished overseas guests were present and were entertained by the President at the Adelphi Hotel.

Other important activities of the Society during the year included the Second Spiers' Memorial Lecture, given by Dr. Robert Mond in June. The subject was Michael Faraday and the lecture was fittingly given at Faraday's lecture table in the Royal Institution. In addition, Professor Müller lectured before the Society in September on the subject of "Passivity."

Institute of Brewing

THE Research work of the Institute of Brewing is mainly concerned at the moment in studying the relative brewing value of malt and hops, both from their fundamental scientific and practical aspects. The value of malt is based largely on the amount of extract it gives and on the activity of the enzymes. The investigations which have been carried out over a number of years on barleys and malts have made it possible to devise methods, based on analysis of the barley, which can be used to predict these two most important factors in the quality of the malt which can be produced from that barley. They should prove of first rate technical importance in enabling a truer estimate to be formed of the brewing value of barley than has hitherto been possible and also provide a reliable method for controlling malting operations. The earlier investigations on hops were directed to obtain the fullest possible information on the nature of the preservative substances contained therein and have led to the formulation of a standard method of analysis intended to help in commercial transactions.

An entirely new method for determining the relative anti-septic value of solutions has been devised, which should have general application in biological research connected with anti-septics and their action on bacteria. It is being applied to an investigation of the changes brought about in the hop anti-septic during wort boiling and fermentation, a question of the highest importance in relation to the stability of beer.

The yeast research is at an early stage—but the preliminary work has led to the discovery that the usual methods for determination of nitrogen in yeast, and possibly in many other substances, is unreliable. Reliable methods have been found which should not only be of use in the contemplated investigations on the nutrient requirements of yeast in fermentation, but they also indicate that the methods at present employed in food analysis and otherwise must be reconsidered.

Institution of Chemical Engineers

DURING the year 1931 the continued progress of the Institution is reflected in an increase of membership of about eight per cent.

At the Annual Corporate meeting, held in March, Mr. W. A. S. Calder was elected President, Mr. Geo. Gray and Dr. W. H. Hatfield were elected Vice-Presidents, and Professor J. W. Hinchley and Mr. F. A. Greene were re-elected hon. secretary and hon. treasurer, respectively. In lieu of giving a Presidential address, the President opened a discussion on chemical engineering education, to which contributions were made by Sir Frederic Nathan, Sir Robert Robertson, Professor Arthur Smithells, Dr. A. E. Dunstan, Mr. C. S. Garland, Mr. J. McKillop, Mr. W. Rintoul, Mr. F. H. Rogers and Mr. H. T. Tizard, truly an indication of the widespread interest in this subject. The Moulton Medal for 1930 was presented at this meeting, to Mr. A. T. King, and the junior Moulton Medal and prize to Mr. L. W. Blundell. The Osborne Reynolds Medal was presented to Mr. J. Arthur Reavell, in recognition of his many services to the Institution since its inception and particularly during his two years in the office of President.

Two public lectures were delivered before the Institution during the year, the first, in March, being by Baron Gian Alberto Bianchi, on "The New Italian Leucite Industry," and

the second, in October, by Dr. E. F. Armstrong, on "Hydro-generation."

No conference was held during the year, but a symposium of papers on less familiar forms of extraction was presented at the Annual Corporate Meeting, as follows:—"Flax Wax and its Extraction," by Dr. W. H. Gibson; "The Recovery of Gum from Fossil Kauri Timber," by Dr. R. G. Israel; "The Extraction of Terpene Chemicals from Waste Pine Woods," by Mr. I. W. Humphrey. These papers were followed by a paper on "The Flow of Gases at High Pressures through Metal Pipes," by Dr. D. M. Newitt. Other papers read during the year were: "The Concentration of Phosphoric Acid by Submerged Combustion," by Mr. C. Featherstone Hammond; "Oils and Oiling, especially in the Textile Industry," by Mr. W. F. Vickers; "The Gas Generator as a Direct Producer of Metallurgical Products," by Mr. N. E. Rambush and Mr. F. F. Rixon; "The Manufacture of Asphalt from Cracking Process Residues," by Mr. F. M. H. Taylor. The paper by Mr. Vickers was read in conjunction with a very successful visit to the Wool Industries Research Association at Leeds. A visit was also paid, on September 11 to the Shipping, Engineering and Machinery Exhibition at Olympia, when members of the Institution were the guests of the Organising Committee. Six meetings were held by the Graduates and Students Section. In May, Mr. H. A. S. Gothard vacated the office of chairman and Mr. F. J. Bailey that of hon. secretary. They were succeeded by Mr. Keith Fraser and Mr. L. W. Blundell, respectively. The President's Reception, held on November 26 at the New Princes' Galleries, again attracted a large and distinguished gathering, some five hundred members and friends being received by the President and Mrs. Calder. The Associate-Membership examination for 1931 followed the lines of those of the two preceding years, and the Examiners, in their Report, noted a continued improvement in the handling of questions from first principles.

All the events of the year have been overshadowed by the severe loss to the Institution in the death, in August, of Professor J. W. Hinchley. Professor Hinchley did more than any other one man to bring the Institution into being, and he served as its honorary secretary from its incorporation in December, 1922, to his death. The many tributes which have appeared in the daily and technical papers testify to the affection in which he was universally held, and to the respect and admiration which his character and work inspired. The memorial service to him, held on October 3 at the Ethical Church, Bayswater, was attended by a great many members of the Institution and of the chemical industry generally. In October the Council appointed Mr. H. W. Cremer hon. secretary, in place of the late Professor Hinchley. Mr. Cremer, who was elected to the Council of the Institution last year, obtained a wide technical experience under Mr. K. B. Quinan during the war in the design and operation of plant for the manufacture of explosives and chemical warfare supplies, and later, as a member of the staff of the Department of Scientific and Industrial Research, he compiled several volumes of the Technical Records of Explosives Supply, which were published by H.M. Stationery Office during 1919 and 1920. He was appointed to the Chemistry staff of King's College, London, in 1920, and has since kept in close touch with the industrial side of the subject. He now holds the position of Director of Chemical Engineering Studies at King's College, for the institution of which course he was responsible some four years ago.

Institution of Petroleum Technologists

THE membership of the Institution which at December 31, 1930, was 1,237 has increased during the year 1931 to 1,272.

Eight general meetings have been held during the year at which papers relevant to the petroleum industry were read and discussed. At the 18th annual general meeting held on March 31 last Mr. James Kewley was elected President for the year and Sir John Cargill, Mr. A. E. Chambers, Mr. T. Dewhurst, Mr. Alexander Duckham, Mr. Arthur W. Eastlake, the late Mr. Robert Redwood and Mr. A. Beeby Thompson were elected or re-elected vice-presidents.

The Institution and the industry suffered a severe loss by the death of Mr. Robert Redwood in September. Mr. Redwood was a Founder Member of the Institution and had been a vice-president since 1924.

During 1931 the Institution has been represented at the Centenary Celebrations in honour of Michael Faraday, the Third International Conference on Bituminous Coal, the Lubrication Congress at Strasburg, and the 11th Congress of Industrial Chemistry at Paris.

A Cancer Research Committee has recently been set up by the Institution to inquire into Mule Spinners' Cancer.

The Committee for the Standardisation of Methods of Testing Petroleum and its Products has continued to hold its meetings. Several new sub-committees have been formed to deal with special subjects, such as the Knock-Rating of Motor Fuels and the Measurement of Oil in Bulk.

Commencing with 1931 the Journal of the Institution is being published in twelve monthly parts, and contains all papers read before the Institution, contributed papers and a very full set of abstracts of petroleum literature and patent specifications.

National Benzole Association (N.B.A., Ltd.).

DURING the past year, the Research Committee of The National Benzole Association has made further progress in a number of problems, which have previously engaged the attention of the Committee. The possibility of preventing during storage the resinification of comparatively crude benzoles by the addition of inhibitors, and of subsequently utilising such benzoles satisfactorily as motor fuel, has now been more thoroughly tested on the technical scale. Fleet trials have been carried out and the result of these so far has shown that stabilised gas works benzole, produced under normal working conditions gives satisfactory results, no gumming of engines having been observed. Further trials on coke oven benzoles are now in progress. Owing chiefly to the discovery that certain types of cracked petrols, possess appreciably higher anti-knock values than straight run petrols, the work of the Committee on the problem of the gumming of motor spirits has recently attracted considerable attention.

Further investigations have been made on wash oils for benzole recovery, such as an examination of the absorptive capacities and stabilities of sulphur treated creosotes. The deterioration of creosote oil and gas oil when used under working conditions for the recovery of benzole from coke-oven gas, has also been carefully investigated, and much information obtained of value to benzole producers. Owing to differences in the design and operating conditions of the plants, however, no comparison was possible from the point of view of costs and efficiencies of recovery with these two oils.

Owing to the growth in the research activities of the Committee, the research staff of The National Benzole Association has now been transferred to a new laboratory at Willesden, and the connection of the Association with the University of Leeds has been terminated. The work previously carried out by the Joint Benzole Research Committee is now being continued by a Committee composed of members of The National Benzole Association.

Society of Dyers and Colourists

MEETINGS, works visits, and lectures under the various sections of the Society have been held in Bradford, Huddersfield, Manchester, London, Leicester, Nottingham, Derby and Glasgow, particulars of which are contained in the Journals issued during the present year.

The Gold Medal of the Worshipful Company of Dyers was awarded in the year 1931 to Dr. H. H. Hodgson, for his paper on "Colour and Constitution from the Standpoint of Recent Electronic Theory."

In connection with the *Colour Index* of the Society, the demand for this work and also for the *Supplement* still continues.

The work on "Standardising the Methods of Testing the Fastness of Dyed and Coloured Materials" is progressing very favourably, and a full report of the work on the different tests should be published shortly.

Society of Glass Technology

IN the course of the year 1931 the Society held nine meetings, four in Sheffield, three in London, one in Newcastle-upon-Tyne, and one in Stourbridge, at which some twenty papers were communicated. In addition, the London Section of the Society met on six occasions. Members of the Society also had the privilege of visiting several industrial plants and other institutions.

Members of the technical committees of the Society visited Germany in May to hold a joint conference with the technical committees of the Deutsche Glastechnische Gesellschaft. In October there was opened in the Science Museum, London, an exhibition to illustrate the most striking developments of recent researches in glass technology. This exhibition was organised jointly by the Society of Glass Technology and the Glass Research Delegacy of Sheffield University. Weekly lectures were given from October 22 to December 17. The *Journal* of the Society for June published a provisional "Standard Test and Specification for the Chemical Durability of 4 oz. Flat Medicine Bottles."

At the annual general meeting, Mr. Edward Meigh, M.B.E., M.Sc., was elected President for the year 1931/32, in succession to Mr. Christopher Wilson. The general treasurer is Mr. Bernard P. Dudding, M.B.S., A.R.C.Sc., F.Inst.P., the American treasurer, Mr. F. C. Flint, Washington, Pa., and the Honorary Secretary, Professor W. E. S. Turner, Darnall Road, Sheffield.

Institute of Chemistry

THE Report of the Council issued in January last showed that there had been a substantial increase in the Register of Members and the present indications are that this rate of increase during the year has been fairly well maintained. The Institute has now a membership of about 6,000, and is probably the largest incorporated body of professional qualified chemists in the world.

Examinations for the Associateship have been held in London and in Manchester, and for the Fellowship, in special branches of work, at the Institute and at various local centres. The scheme of examinations for the award of National Certificates, in conjunction with the Board of Education in England and Wales, and with the Scottish Education Department, continues in satisfactory operation, and it is hoped that, before long, examinations for these Certificates will also be held in Northern Ireland. The Carpenter Committee has recommended that the Ordinary National Certificate should be accepted as a qualification for ancillary staffs of Government Departments.

When the Poisons and Pharmacy Bill was before the last Parliament, the Institute endeavoured to secure representation on the Poisons Board, and a definite understanding with regard to the use of the title "chemist" by chemists who are not necessarily pharmacists. Simultaneously, the Council made the proposal to petition for a Supplemental Charter to confer upon the members of the Institute the exclusive right to use the title "Chartered Chemist." On the matter being put to a postal vote, a large majority of the members voting, was found to be in favour of the proposal, although criticism arose, both from a section of the members and from other bodies who considered that their interests might be affected. As the Poisons and Pharmacy Bill was not proceeded with before the change of Government, and it is uncertain when it will be re-introduced, these matters have had, for the present, to be left in abeyance.

In June, the Institute published statistics compiled from the returns received from 3,828 members in answer to an inquiry into their remuneration. Although these returns indicated that a large proportion of Associates—62 per cent.—were receiving remuneration between £250 and £500 per annum and only 30 per cent. of the Associates were receiving more than £500 per annum, the returns from Fellows showed that over 42 per cent. were receiving between £500 and £1,000 per annum, and a further 35 per cent. were either employers or in receipt of remuneration exceeding £1,000 per annum. These returns were for the British Isles; the returns from Overseas Dominions and Abroad were naturally higher. It is felt that the publication of the figures provides very useful information to chemists and employers, and also to those who are responsible for the education of students who intend to pursue chemistry as a profession.

As could only be expected in the present state of industry, the number of vacancies for chemists notified on the Appointments Register of the Institute, showed a marked falling off during the year; but notwithstanding this circumstance and an increasing membership the number of members known to be without appointments is still under 3 per cent.

The Benevolent Fund Committee has met all appeals as generously as possible with the funds at its disposal, but finds

that in the present circumstances a special endeavour must be made to secure contributions in order to meet the needs of the coming year.

In conjunction with the Association of British Chemical Manufacturers, the Institute has formulated a suggested Contract of Service for Chemists. The Joint Committee of the two organisations recognised the existence of many cases wherein such a contract was unnecessary, and felt that it would be impossible to formulate a Contract which would be generally applicable. The Committee suggested, however, that the form would be found to cover comprehensively the terms and conditions which might be incorporated in such contracts, having regard to the interests both of chemists and of employers. The suggested form is available for the use of members and of employers who may wish to refer to it when such contracts are under consideration.

Among other matters with which the Council has been concerned during the year may be mentioned the Report of the Carpenter Committee, the fumigation of ships, the Report of the Fertilisers and Feeding Stuffs Advisory Committee, and the preparation of a Memorandum submitted to the Departmental Committee on Food.

The rooms in the premises of the Institute which were formerly occupied by the Ministry of Munitions and later by the British Photographic Research Association have been converted into a new meeting and examination hall. The attendances at lectures of the Institute and at meetings of the London Section and other bodies have greatly increased in recent years, and the accommodation previously provided for such meetings, in the Council Room and Library, has proved to be somewhat inadequate.

The seventeen Local Sections of the Institute which have been formed in important centres in Great Britain and Ireland, at the Cape, in New Zealand and in Malaya, have continued active, and many interesting papers have been read before them.

The Institute has published the seventh edition of the "List of Official Chemical Appointments," and, as a separate monograph, Dr. J. F. Tocher's lecture on "What is Probable Error."

The Fourteenth Streatfeild Memorial Lecture was given by Dr. J. Vargas Eyre, whose subject was "Recent Advances in the Fermentation Industries" and the Third S. M. Gluckstein Memorial Lecture was given by Sir Frank E. Smith, K.C.B., F.R.S., and entitled "The Chemist and the Community."

The Meldola Medal for 1930 was awarded to Dr. R. P. Linstead, of the Imperial College of Science and Technology, and the Sir Edward Frankland Medal and Prize for Students, to Mr. Geoffrey Broughton, of the Royal Technical College, Salford.

Dr. Barnett Levin has been elected the Second Pedler Research Scholar and is working on "The Determination of very small quantities of Calcium and Magnesium under varying conditions," in the Chemistry Department of Guys Hospital Medical School, under the general direction of Professor C. S. Gibson, F.R.S.

Oil and Colour Chemists Association

THE Oil and Colour Chemists' Association, of which Mr. Noël Heaton was again elected to the office of President, has continued to make considerable progress during the past year. It is very satisfactory to note that the yearly increase of membership of this Association, which has been reported in former years, still continues. Both in the quality and quantity of the Papers read before the Association and also in those communicated, progress has been maintained. It is thus evidenced that interest has not abated in the scientific development of the manufacture of paint and varnish.

Under the Chairmanship of Mr. G. A. Campbell the Manchester Section of the Association continues to uphold the traditions of the Section.

The opening meeting of the Session 1931-32 was held at the Palace Hotel, Bloomsbury, London, which was preceded by an informal dinner. The Annual Dinner was held at the Connaught Rooms, London, being followed by a dance, on March 27, 1931.

The Section of the Annual Reports on the Progress of Applied Chemistry, (1930) dealing with Paints, Pigments, Varnishes and Resins was again produced by members of the Association under the Editorship of the Honorary Editor (Mr. A. A. Drummond) of the Association's Journal.

Chemical Inventions in 1931

By our Patents Correspondent

Brief indications of the trends of invention in a few of the more important branches of chemical industry, as represented by the Specifications published during the year, are outlined in the following article.

CYCLES of industrial expansion and contraction have not in the past had any very notable effect upon the output of inventions as measured by the number of applications for patents in this country. Any slight effect observable has usually indicated some quickening of inventive activity in periods of industrial depression. This was perhaps only to be expected. Presumably, in prosperous times larger funds are available for research, but whereas reasonable profits are obtainable from well-established processes there is no overpowering incentive to cheapen the cost of production by the discovery of new processes or by improving the old. In the depression which follows, however, that incentive becomes insistent and the inventor's services are again in greater demand.

It is perhaps one indication of the abnormal character of the present depression that until the present year invention has been greatly stimulated by the progressively difficult conditions under which all industries have been working. There were, for example, approximately 20 per cent. more applications for patents received by the British Patent Office during the black year 1930 than during the comparative prosperity of 1925. The present year has seen some retrogression, however, applications at the time of writing being some 8 per cent. below the number for the corresponding period of 1930.

Effect of Conditions in Germany

Turning from invention over the whole field to invention in the chemical industries, this retrogression is found to be much greater, and this is probably to be explained with reference to the very difficult financial conditions in Germany.

In this connection the I. G. Farbenindustrie Akt.-Ges., of Frankfurt-on-Main, may be taken as essentially representative of German chemical industry as a whole, and it is significant that in comparison with 326 applications for British patents in the third quarter of 1929 that organisation made only 120 applications during the corresponding quarter of this year.

International Distribution of Patent Interest

Of British chemical patents at present in force less than one-third appear to be held in this country, and of the other two-thirds German interests appear to hold more than all other foreign interests combined. Recent years have seen a remarkable increase in the number of chemical inventions originating in the United States. They are, however, concerned for the most part with the oil refining, lacquer, and rubber industries. Inventions relating to dyes, dye intermediates and dyeing processes are still of German, British or Swiss origin, with very few exceptions. Few chemical inventions originate in France, still fewer in Holland, Belgium or Italy, and the number from all other countries combined is insignificant.

Cellulose

That inventions in connection with cellulose and its derivatives have been very numerous is the predominant impression on glancing through the titles of the recently published specifications. The national origins of such inventions also appear to be more widespread than in any other branch of the chemical industry. All the countries mentioned above and one or two others are represented. It is also evident

that the recent rapid developments in the technique of the artificial silk industry have had far-reaching reactions on all the other arts based on cellulose products especially on the manufacture of cellulosic lacquers and other coating compositions, of various plastic compositions and adhesives and of celluloid and like films. If one may judge from the large proportion of the inventions relating to the cellulose ethers these are coming to have a technical importance comparable with that of the cellulose esters and the regenerated celluloses in the recent past. In the artificial silk industry itself there are, in addition to the usual number of improvements in wet and dry spinning processes and apparatus, many inventions of processes for incorporating in the spinning solutions or applying to the freshly spun filaments various chemical substances adapted to modify the dyeing properties of the finished yarn or fabric or to enhance or decrease its lustre or even in some cases to impart a phosphorescent finish.

Dyes

The Colour Index (1924) enumerates some 1,300 chemical compounds commercially available as dyestuffs and offered to the colour user under some 11,000 trade names. The Supplement (1928) thereto adds some 4,000 more such trade names and the activities of inventors in this field must have added many more during the past three years. To the uninitiated this at once suggests that there may be scope for some drastic measures of rationalisation in the dyestuffs industry. However that may be, the manufacturers continue to make strenuous efforts to meet the demand for more and better dyes, and as in most recent years the inventions relating to the vat dyes of the anthraquinone series and the azo dyes have been the most numerous. The present rigorous demands as regards fastness to light in textile dyeings and in coloured lacquers probably account for the large volume of research on the anthraquinone vat dyes. Inventions relating to dyes of the benzanthrone series and especially the halogenated derivatives of benzanthrone, violanthrone and isoviolanthrone have been particularly numerous in this field during the year.

Among the azo dyes comparatively few inventions have been concerned with improvements in the dyeing of wool or cotton with the exception of a number relating to new ranges of components for use in the production of azo dyes on the fibre, mainly on cotton, wherein the coupling components are of the Naphthol A.S. type. Similar ice colour dyeings have been suggested in which the arylides of 2:3-oxynaphthoic acid are replaced by arylides of hydroxyphenanthrene-*o*-carboxylic and hydroxycarbazole-*o*-carboxylic acids. Numerous inventions have been concerned with the production of azo dyes and of complex metal (copper, chromium, etc.) compounds thereof suitable for incorporation in varnishes and lacquers.

Dyeing Acetate Silk

Among the azo dyes for acetate silk are some containing the anthracene (other than the anthraquinone) nucleus, various complex metal compounds of azo dyes having a mordant character, and numerous quite simple monoazo dyes such as *p*-hydroxybenzene-azo-1-phenyl-3-methyl-5-pyrazolone, *p*-aminobenzene-azo-1-phenyl-3-methyl-5-pyrazolone and benzene-azo-1-(oxyalkyl)-amino-naphthalenes. Dyestuffs of other types for this material include galloxyanines applied in the leuco form and developed by oxidation, free leuco compounds of vat and sulphur dyes in general including substituted and unsubstituted amino-anthraquinones, similarly applied and developed, 4-sulphamic acids of 1:8-naphthalimide derivatives, and amino-anthraquinones substituted in the nucleus by a carboxamide group and one further auxochrome group.

Dyeing Regenerated Cellulose Silks

Dyes for artificial silks of the viscose type have been, almost without exception, either azo (usually diazo or poly-azo) dyes or their complex metal compounds. Such azo dyes are not necessarily new *per se*, as a useful and valid invention may be based on the discovery that dyes long known to be applicable to cotton or wool are capable of yielding fast level dyeings on viscose. Both the azo dyes and the complex metal compounds are usually applied in substance. These dyes, with the exception of some derived from aminoanthrahydroquinone disulphuric esters, are invariably sulphonated. A few inventions, however, relate to the production on regenerated cellulose fibres of unsulphonated azo dyes having as coupling components arylides of 2:3-oxynaphthoic acid.

Hydrocarbons

Inventions relating to the destructive hydrogenation of coal, oil, etc., have been less numerous than in some recent years and have been concerned mainly with improvements in the details of the known processes, such as the regeneration or purification of the hydrogen-containing gases at some stage in the cycle of operations or the control of the heat economy in the cycle. Improvements relating to the use of catalysts in special physical form, to the elimination of poisoning of the catalyst, and to a combination of the hydrogenation with a simultaneous production or incorporation of anti-knock constituents for motor fuels are also to be noted.

The conversion of methane, and other hydrocarbons of the paraffin series or gases containing them into acetylene is the subject of numerous inventions having for their ultimate object the synthesis of acetic acid and acetaldehyde from economically produced acetylene. These inventions include various improvements in the known electric discharge processes, heating of the hydrocarbons at temperatures up to 2,000° C. in presence of steam, hydrogen chloride or other diluents or of halogens or other catalysts, and the incomplete combustion of the gases in presence of elementary silicon.

Synthetic Rubber

No summary of the year's inventions in connection with the chemistry of hydrocarbons would be complete without some brief reference to the large number, emanating almost entirely from the I. G. Farbenindustrie Akt.-Ges., relating to the polymerisation of butadiene, isoprene and other diolefines for subsequent emulsification and coagulation into synthetic caoutchoucs. In most cases the intermediate or final products are described as suitable for use as coating preparations, for the making of films and artificial silks, and for incorporation in lacquers and adhesives. It is of interest to note that inventive activity has continued in this field for several years past in spite of the low price of natural rubber.

Metallurgy

In the metallurgical field there have been very few inventions relating to the extraction of metals from their ores and it is of interest to note that these few have been concerned mainly with the extraction of zinc and aluminium. On the other hand inventions relating to the production of new alloys have been exceptionally numerous. Thus the electrical industry is making heavy demands on the metallurgist for alloys of approved constitution and having the prescribed qualities required for electric resistances and heating elements, for armouring cables, for filaments of electric lamps and thermionic discharge valves, and for loading inductances. Much attention is also being given to the production of iron alloys resistant to atmospheric corrosion and to acids, of alloys for solders, and of alloys for lining bearings.

Netherlands Nitrogen Foreign Trade

The development of synthetic nitrogen production in the Netherlands has led to an expansion in export sales. Netherlands shipments of ammonium sulphate during the first nine months of 1931 totalled 176,633 metric tons, which represents a remarkable growth when compared to the 64,977 tons exported in the corresponding period of 1930. Imports of nitrogenous fertilisers in the early months of 1931 were considerably less than the receipts for the previous year. The position was reversed during September by large receipts of foreign materials, principally German. September importations of ammonium sulphate of 42,353 tons brought the total for the nine months to 49,608 tons of which 43,978 are credited to German sources. Similarly, the September receipts of 25,409 tons of calcium nitrate brought the nine months' total to 56,322 tons. While Norway was the principal source of supply for the nine months' period, Germany furnished 60 per cent of the September importations.

Increase in United States Drug Manufactures

The value of drug manufactures produced in the United States in 1928 was 15 per cent. above 1927, according to final figures just published by the Bureau of the Census Department of Census, Department of Commerce. All branches of the industry advanced: Druggists' preparations, 15 per cent.; proprietary medicines, 15 per cent.; proprietary compounds, 21 per cent.; perfumes, cosmetics, and other toilet preparations, 13 per cent.

BROOMWADE

ROTARY COMPRESSORS & EXHAUSTERS

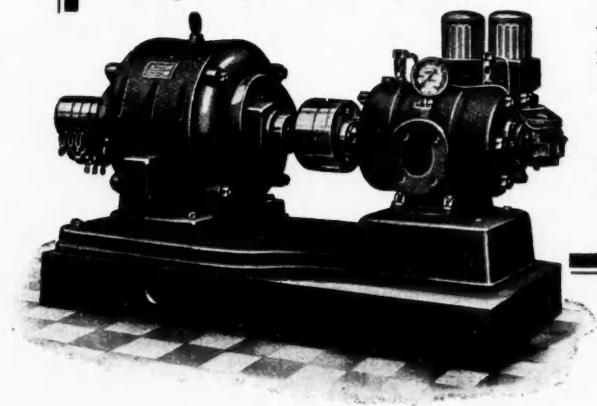
MANUFACTURED in a range of sizes from 6 to 1200 cubic feet per minute capacity, the "Broomwade" Rotary Machine is super-efficient, and its running speed and low starting torque enable it to be direct coupled to comparatively high speed squirrel-cage motors or other forms of driving units.

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materials and workmanship are cardinal features which are strikingly apparent to the Plant Engineer.

Among this range there is a machine particularly suitable for your special needs—a machine capable of giving under the most arduous conditions a full measure of efficiency and reliability, and a machine embodying the results of 30 years' exhaustive effort in the design, manufacture and installation of Air Compressing Machinery.

The name "Broomwade" has become the standard by which Air Compressing Machinery is set; that is why Engineers the world over specify "Broomwade" when conditions call only for the Best Possible.



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HIGH WYCOMBE

The Voice of the Industry

We give below extracts from reports on the year which have been received from well known firms in the chemical and chemical engineering industries.

Aluminium Plant and Vessel Co. Ltd. Wandsworth, London

THE year has been noteworthy for a considerable increase in the demand for autogenously welded plant in non-ferrous metals. It has therefore been necessary largely to increase the productive capacity of the works, and the call of the chemical industries for still larger units of plant has been met by the erection of spacious shops specially designed for the manufacture of large distillation columns, reaction vessels, etc. New chemical and metallurgical research laboratories have also been erected and equipped, and a technical laboratory has been provided for experimental work on a semi-technical scale.

The A.P.V. patented type of heat exchanger, which has proved such a success in the liquid foodstuffs industries, is proving of increasing interest to chemical manufacturers and many machines are now working on acetic acid, varnish oils, and cooling schemes involving the use of sea water. This machine is of the plate type but no provision for "dirt factor" is necessary as all surfaces are easily and readily cleanable by unskilled labour, which is not the case with the more usual tubular heat exchanger. Another important advantage of this machine is its elasticity of output as plates can be added or removed in a few minutes to suit variations in duty if required by special circumstances. Incidentally very high rates of heat transfer can be obtained with this machine due to the special design wherein the turbulence is increased by the shock effect of reversals of flow after apparently short lengths of flow through the channels. Based on this type of heat exchanger a new type of evaporator is being developed wherein both sides of the heating surface can be readily cleaned. Evaporators built up on this system are particularly suitable for dealing with viscous liquids and liquids where low temperature differences are necessary, or which deposit scale. Several special process vessels over 16 feet in length, with approximately 1,000 heating tubes, and with shells $\frac{3}{8}$ in. thick have recently been constructed to withstand a test pressure of 100 lbs. hydraulic for one hour. Many large copper vessels of the tubular type and large diameter column stills have been constructed with hammered autogenous welds to meet specially severe conditions where neither brazing nor soldering would be permissible. Considerable development has also taken place in the construction of autogenously welded vessels, such as solvent extraction apparatus, jacketed mixing pans, and reaction vessels in monel metal and nickel. Several complete varnish and oil boiling kitchens have been designed and equipped, complete with fume removal plant, oil-firing systems, kettles and subsidiary machinery.

The British Drug Houses, Ltd., London

THE year 1931 was one of difficulties so great as to be unexampled from some points of view. Since the war we have experienced—in 1921, the great slump; in 1926, the general strike and the six months' coal strike; in 1930, deep industrial depression. The gloom of 1930 deepened in 1931, and this—the year under review—has given us its own special adverse features. The buying capacity of practically every export market has seemed to be "sick of a fever," while competition in the home market, due to overproduction, has been at least as bad as ever. The last two or three months have shown some improvement.

British Industrial Solvents, Ltd. London and Hull

THIS Company, like others in the chemical industry, has suffered from the trade depression, but research and development have continued, and plant and process improvements have been effected. With an improved economic situation, prospects are distinctly brighter, and the Company is well equipped to take full advantage of the greater opportunities which it anticipates in the coming year.

Thomas Broadbent and Sons, Ltd. Huddersfield

WE have found business during the current year to be rather worse than it was during the previous year. The home market has been very flat indeed, but it is pleasing to record that our overseas trade has been considerably better. Since October, we have booked several large orders for abroad, and the home trade has been very much brighter. We confidently expect a general all-round improvement in industry during the forthcoming year.

John C. Carlson, London

IN spite of the bad industrial conditions experienced during the past twelve months good progress has been made in the various branches of the chemical industry. Prospects for the coming months, too, appear to be good, and promising inquiries have been received in anticipation of the long overdue trade revival. In particular, outstanding success has been met with in the vegetable and edible oils and sugar industries. As far as the oil industry is concerned, plant has been erected in some of the most prominent refineries, for the final clarification of refined deodorised oils, and has given consistently good results. In the sugar industry filters have been installed in both beet sugar factories and refineries, dealing with both beet and cane sugars. As regards beet sugar, the most prominent feature has been the installation of plants to deal with limed raw juices. The raw juice, direct from the diffusion batteries, is given a preliminary defaecation with about 0.2 per cent. of lime. The resultant mixture is filtered through the Seitz' filters with tremendous success. The advantage of the process is the decreased formation of molasses.

Chemical and Metallurgical Corporation, Ltd., Runcorn, Cheshire

IN spite of the abnormally depressed trade conditions of the year, we have increased both our tonnage output and our net cash turnover. Our plants, without exception, were especially designed for the production of the various chemicals in which the Company is interested. In addition, as the Chairman announced in his statement regarding the activities for 1929, a further £100,000 was set aside to cover the cost of a re-organisation and modernisation where this course had become necessary. The result is shown in our opening statement. We have also undoubtedly been assisted in expansion by having acquired a reputation not only for producing goods of exceedingly high quality, but for maintaining this high standard unvaryingly.

W. Edwards and Co., Denmark Hill, London

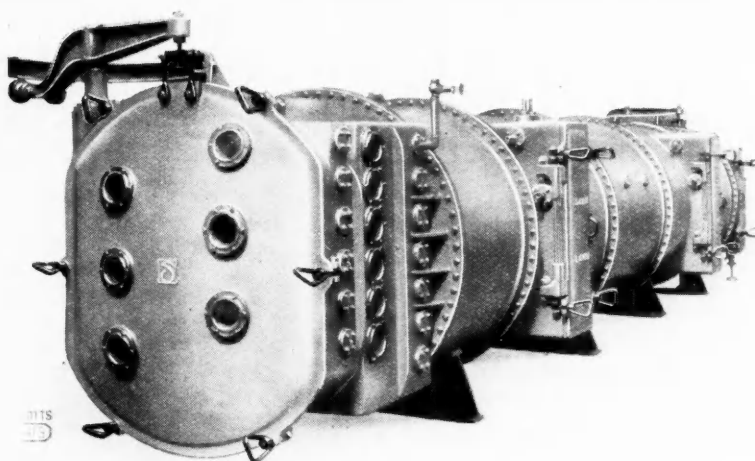
DURING the past year we have introduced a number of new high speed pumps giving exceptionally high vacua and making possible the simplification and speeding up of many manufacturing processes. The well-known "Type A" and "B" pumps supplied by us are in constant use in factories all over the country. The new single stage A2 and A3 pumps have free air capacities of 300 and 450 litres respectively as against 85 for the type "A1" and giving a vacua of 0.01 mm., as against 0.02 for the former type "A" thus combining very much increased capacity with a vacuum improved by 50 per cent. The "Hyvac" series of pumps also supplied by us are very widely used and the latest addition is the Hypervac with a capacity of 210 litres per minute and giving a vacuum of 0.00005 mm.

W. H. Feltham and Son, London

IN spite of the very difficult conditions prevailing in the chemical industry during 1931, our trade in cotton liners for sacks, barrels, etc., has considerably increased, and

THE SCOTT

PATENT COMBINED VACUUM BAND DRYING AND GRINDING PLANT



Continuous in Operation

Drying and Grinding effected under High Vacuum

Ideal for Malt Extract, Malted Foods, Milk and Milk Products, and many Delicate Materials, demanding low temperature and perfect hygienic conditions

Demonstration Plant Available

GEORGE SCOTT & Son (London) Ltd.
ERNEST SCOTT & Company, Limited

Bradfield Road, Silvertown, London, E.16. Glasgow Office 19 Waterloo St., C.2

manufacturers of chemicals appear to be realising the benefit of using cotton liners to keep their products in clean condition and free from contamination with the outside package. We anticipate 1932 will show a still further increase in the sales of these liners, as prices are now extremely low.

Foster Instrument Co.,
Letchworth, Herts

OUR apparatus, mainly devoted to electrical temperature measurement, serves practically every class of manufacture and is therefore, naturally, widely disseminated. As new instruments are only required when new plants are installed or when an old plant is reorganised, our business in new pyrometers constitutes a "barometer" of manufacturing conditions. The advent of the new Government was followed almost immediately by an increase in business, which has continued. Although there have been some increases in the cost of our material, we have not yet increased our prices, nor have we any present intention of doing so. Export business has not grown as we hoped, notwithstanding the encouragement there should be due to the lower international value of the pound, but we have no doubt that some of our increase in home business is due to increased export business experienced by home customers for our instruments. Taken as a whole, we think that the present conditions are most encouraging and we have a hope that the dismal forebodings which were freely voiced some time back will be found, as often before, not to materialise.

Wm. Gardner and Sons (Gloucester), Ltd.
Gloucester

CONSIDERING the difficult trading conditions that have prevailed throughout the year we have been able, owing to our many specialities in grinding, sifting, drying and mixing machinery for all kinds of powders, to keep our works fully occupied and without discharging any employees. We have opened up new departments during the period and have executed many foreign orders. It is difficult to say how the new trading conditions will turn out. We have many orders on our books, and some valuable inquiries, and when matters settle down to more normal conditions, we believe that we shall have a busy time in front of us to execute orders for the new manufactories which are opening up in this country, and for those restarting branches which, owing to unfair conditions in the past, have for a long period been closed down.

General Chemical and Pharmaceutical Co., Ltd.
Sudbury, Middlesex

DURING 1931 we have been engaged in further equipping our new works at Sudbury, Middlesex, and in improving methods of production of some of our staple products, the result being that, in a number of instances, it has been found possible still further to increase the already high degree of purity of the products and to obtain increased output. A considerable amount of research work has been carried out with the object of improving existing processes and also with the object of producing various new products commercially. It is anticipated that, within a short time, we shall be ready to place several of these new chemicals on the market, including a reagent which we believe will prove of considerable value in organic synthesis and also a reagent which will facilitate, to a marked degree, certain analytical work. Despite the difficulties of the year production has been successfully transferred to our Sudbury works and the quantity of our products sold shows a substantial and gratifying increase compared with last year. Manufacturers are calling upon us to an increasing extent to provide their requirements as regards chemicals to special specifications, and, having regard to the flexibility of our new plant, we are looking for considerable expansion, and face 1932 with confidence.

Kemball, Bishop and Co., Ltd.
Bromley-by-Bow, London

CONDITIONS during the past year have, of course, been difficult under the world slump, but with the suspension of the Gold Standard, export inquiries began to be more numerous, and our sales abroad have undoubtedly increased. During the past year we introduced the manufacture of lithium carbonate and lithium hydroxide, two chemicals not hitherto

made in this country. Their use is not large, but it is our view that British manufacturers should to the fullest extent commence the production of articles which are imported, even though, as in the case of lithium, the volume of business is not large.

Meldrums, Ltd.
Timperley, near Manchester

WE have had a steady demand for our "Meldrum metal" specialities during the past twelve months. These consist mainly of our new design of acid pump, which gives freedom from leakage at the gland; pipes, cocks and valves, and other parts connecting with these installations. We have also experienced a steady demand for our steam jet apparatus, exhausters, vacuum apparatus, etc., some being in our "Meldrum" acid resisting metal, and others in cast iron or regulus metal. In our Destructor Department we have completed numerous installations of destructors at private works and for municipal authorities. Many of these are for steam raising or for heating water for buildings and other industrial purposes. We have also experienced a good demand for our forced draught furnaces and mechanical stokers, many of these being to deal efficiently with the black smoke problem, and at the same time to utilise cheaper fuel and get increased steam supplies from existing boilers.

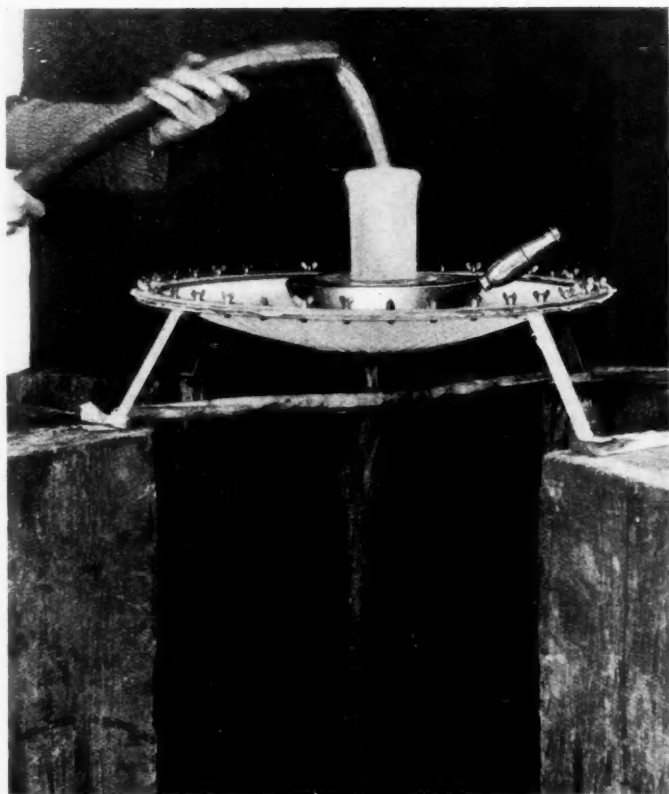
Mining and Industrial Equipment, Ltd.
London, Derby and Erith

INQUIRIES and sales during the past year show a falling off in comparison with the previous year, but new business has only decreased by about 9 per cent. This merely indicates that existing companies have curtailed production. Since coming off the Gold Standard, however, the affect has been to encourage progressive manufacturers to remodel their plants. The inquiries from abroad in different branches of chemical engineering have materially increased and exports from this country have been made to certain continental countries for the first time on record. There appears to be two reactions at the present time—the progressive line in which manufacturers are taking this opportunity to scrap obsolete plant and invest idle money in up-to-date equipment, and the other angle of curtailing production and decreasing staff with the idea of cutting expenses to the minimum and awaiting better time. There is no doubt that the progressive policy will win, as many of those plants which are now using obsolete machinery must either re-equip or close down. At the present time, money wisely translated into plant is a greater asset than money lying in the bank. There is every indication that the coming year will enable producers in this country to establish markets in fields they have not before entered.

Nordac, Ltd.
Harrow, Middlesex

THE past year's work of this firm has consisted of steady development and new methods of applying rubber to chemical work and designing new forms of chemical apparatus. The lining of tanks and vessels with the uncured rubber by the Nordac process is growing rapidly so much so that the large rubber concerns are studying chemical work and in consequence there is now fair competition in this field. The problem of adhesion of rubber to various materials is still to be solved especially to existing plant which is generally damp, and often with acid moisture. The pickling processes connected with metal coating industries such as galvanising are at last appreciating well designed plant. This firm has developed a robust rubber lined tank for use in connection with mechanically operated plant. The need for careful measurement of acid to these tanks is seen and the Nordac patent meter is being installed. The firm's screwed hose fittings which is claimed to be a superior product to ebonite promises to be in demand for the coming year. At the moment a large bleach works is being piped throughout with special hose and these patent fittings. No iron fastenings are to be used in this work. Several rubber concrete tanks have been erected. The properties of rubber concrete for tank construction are gradually being appreciated. It is superior to wood in that it cannot shrink or warp and is as flexible and permits the use of operations between wide ranges of temperature. The use of rubber in concrete is interesting road engineers, and development along these lines is being pursued.

Achievement :



Photograph of a demonstration of the immense wet strength of No. 54 Filter Paper. It should be noted that the paper is merely clamped in the suspending ring, being entirely unsupported otherwise.

To the exceedingly comprehensive range of Filter Papers produced by W. & R. Balston, Ltd., of Maidstone, have been added further triumphs,

WHATMAN No. 54.

A new and outstanding grade of great possibilities.

Its special features are :

1. Filters Rapidly.
2. Has immense wet strength.
3. Precipitate can be scraped off and the paper used several times.
4. Will withstand Caustic Solutions up to almost any strength.

WHATMAN No. 52

Has all the characteristics of No. 54 quality, but is slower filtering and retains finer precipitates.

A revised descriptive booklet and price list has recently been issued, giving many useful notes on the uses and special application of the various WHATMAN GRADES.

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WHATMAN
High Grade FILTER PAPERS

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H. REEVE ANGEL & COMPANY, LTD., 9, BRIDEWELL PLACE, LONDON, E.C.4.

The spraying of rubber is now a normal process of this firm and several large jobs have been done with this method, such as the lining of large water tanks, water softening plant, flues for washed furnace gases, and sugar plant. The firm's rubber emulsions are interesting the cork and paper industries, and one firm uses the material for 3-ply wood manufacture. One interesting development is the supersession of the celebrated German earthenware by rubber. The tiled lined vat will soon be a thing of the past. A suitable rubber cement is needed to make a good job. A number of these tiled vats have been cemented with rubber and broken earthenware vessels of large size have been repaired and made leak-tight. The firm has brought out two new types of valves, for acid work, and research on an all rubber rotary filter is almost completed. The prospects for 1932 are good. Business is expected for pickling, electroplating, dyeing, sugar and food industries, in fact all industries where acid corrosion abounds.

**Redler Patents,
Sharpness, Gloucestershire**

WE have found in the last year, that the Redler system of conveying in bulk has expanded in many directions. The depressing trade conditions which prevailed over most of the year have, in our case, been largely counteracted by the increasing popularity of *en masse* conveying. The British Industries Fair at Birmingham, last February, where we had a large exhibit, brought in an unprecedented number of inquiries, not only from home buyers but also from those abroad. Many of these were slow to materialise owing to bad trade everywhere and we received letters repeatedly postponing improvements for this reason. For a long while, however, we have been busy with many orders continually on hand, which keep our works at full time and for periods, on overtime. A remarkable feature in our trade of late has been the great increase in direct inquiries from different European countries. These, no doubt, are in part due to the depreciation of sterling, and if we are not, as a country, too eager about the Gold Standard, we have little doubt that, when the deplorable effects of the recent dumping have evaporated, this depreciation in sterling will act in favour of trade generally.

**Robinson Brothers, Ltd.,
Birmingham**

IN addition to the old-established business of the manufacture of horticultural and agricultural manures, insecticides and disinfectants, we now manufacture piperidine of exceptional purity and a large range of piperidine derivative accelerators for use in the rubber industry. These accelerators, marketed under the title "Robac" brand, are of a very active type, and offer to the rubber industry considerable scope for variation and control of conditions and vulcanisation at prices competitive with foreign material. New products are constantly being evolved and developed for stock. By our present efforts and further research we hope to assist in rendering the United Kingdom independent of imported accelerators of this type.

**George Scott and Son (London) Ltd.,
Silvertown, London**

OUR experience during the year under review is probably in line with that of other engineers, and certainly of most chemical engineers. We opened the year with a reasonable amount of work carried forward, but new work tapered off, and from the spring until the results of the General Election were known firms requiring new plant obviously held their hands awaiting developments. Since that time, however, business has unquestionably improved appreciably and we close the year with a gratifying order list.

There is obviously a growing confidence abroad, as indicated by the foreign orders we have secured during the past six weeks, and there are indications on every hand that we may hope that this position will be maintained and possibly improved upon in the coming year. The situation which was created by the war, and which resulted in many general engineers having to build chemical process plant which they were not themselves capable of evolving but which were constructed to drawings furnished by Government Departments

and chemical manufacturers, to some extent remains. This relates more particularly to plants of standard design calling for no great originality or wide chemical knowledge. During the last few years it has been evident to us that we must be more and more dependent upon our ability to evolve new processes and design plants for processes evolved by our clients. This view is fully confirmed by the plants we have at present under construction, practically all of which embody novel features and patent designs. These plants relate particularly to vacuum and hot air drying, evaporating plants of our patent forced circulation type, and solvent extraction plants embodying many new features. We are already finding that the import duties announced and anticipated will prove of material benefit to chemical engineers in this country. We look forward to the new year very hopefully.

**Silica Gel, Ltd.,
London**

THE poor trade conditions during 1931 have held up progress in strictly chemical engineering fields, though there have been continued orders from the electrical industry for Silica Gel plants to be used in manufacturing processes. Plants have been supplied, however, to recover ethyl acetate and alcohol from the manufacture of artificial leather; ether and alcohol used in the manufacture of explosives; benzene and also alcohol containing heavier hydrocarbons. These plants have operated very satisfactorily, and the recovery has been well over the guaranteed figure. The drying of oxygen has also been a productive field, plants having been supplied to dry this gas before bottling to extremely low moisture contents. In most of these the gas is handled at varying pressures during operation, the range being anything from 0 to 250 atmospheres pressure. During the last few months of the year there have been signs of greater willingness to lay out capital, and there are prospects of further orders for the recovery of acetone and alcohol from various processes. Air dehydration, particularly for the Tropics, is arousing greater interest. One interesting example of this is the plant recently exported for the conditioning of a University Library. In addition to the continued activity in the electrical industry, there also appear to be prospects for further orders for dry blast and various gas dehydration plants.

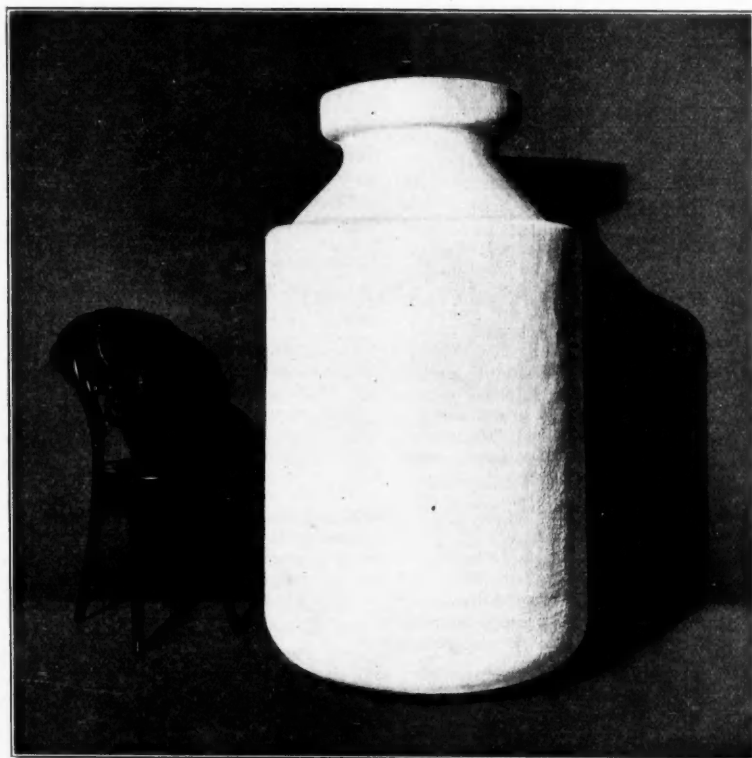
**Staveley Coal and Iron Co., Ltd.,
Chesterfield**

So far as our chemical department is concerned, the year was dull and uneventful during the first eight months or so. Competition, however, was keen. In September the prices of some commodities improved and during October and November there has been an appreciable increase in the demand, whilst some plants which were previously working under reduced output are now working at full pressure. This year has also seen a considerable falling off in the demand for sulphuric acid, due to the uneconomic price that has been ruling for some months for sulphate of ammonia. Many gas works and coke ovens are now wasting as much of their ammonia as they can. The increase in the demand at the present time comes chiefly from the textile, paper, dyestuffs and chemical trades. The departure from the Gold Standard in this country seems to have brought a bigger share of the trade to this country. If tariffs are imposed there seems to be every likelihood of trade in this country being better during 1932 than during 1931. The extent of this improvement, however, is difficult to determine as it is impossible to say what will be the effect of the present financial crisis in Germany. The cancellation of War Debts would clarify the outlook somewhat.

**Thomas Tyrer and Co., Ltd.,
Stratford, London**

WE have carried on business for some time past under very difficult conditions, but have by hard work and perseverance succeeded in getting through the year fairly successfully. Conditions during the past year, for instance, have been very difficult indeed, but with the present change in Government and the economies which are to be effected, we hope that the condition of trade will gradually improve and unemployment will be decreased. It certainly seems that there are slightly more prospects as far as trade is concerned.

VITREOSIL CONTAINERS



Improvements in manufacture have made possible the production of containers or reaction vessels of large size. The picture shows one of 108 gallons capacity. It is 4ft. 6in. in height overall, and 2ft. 6in. internal body diameter.

THE THERMAL SYNDICATE LTD.

VITREOSIL WORKS, WALLSEND-ON-TYNE

LONDON DEPOT : THERMAL HOUSE, OLD PYE STREET, S.W.1.

Whiffen and Sons, Ltd.,

Fulham, London

THE year has been outstanding as a year of depression, curiously alternating with an optimism which unfortunately has been too often short lived. However, we enter the last phase of the year with renewed hopes justified by the strongly expressed intention of the country to have fair trade at last. For us the year has been a long drawn out fight with foreign competition, particularly in the bromide market, but we have the satisfaction of feeling that we have held our own well, and with the added stimulus of Empire bromine and, we hope, adequate protection, we are confident of seeing the industry placed at last on a sound and generally satisfactory basis. We have been active in research and have added to our manufactures some valuable tin compounds such as methyl stannic iodide, tin sulphur iodide, and methyl tin lactate. Other interesting additions are Kurchine hydrochlor, sulphate and phosphate; also the double salts of bismuth and iodine with Kurchine. We have also perfected an alternative to ordinary gelatine under the trade name of "Gelozone"; this product is supplied in a dry powder which will keep indefinitely and is prepared from Carrageen, of which supplies are practically inexhaustible.

Williams (Hounslow), Ltd.,

Hounslow, London

THE year opened with a message of hope for the dye manufacturing industry inasmuch as the Dyestuffs Act, after a stormy passage through the House of Commons, got happily anchored to the Expiring Laws Continuance Act and found a haven for a further twelve months. Unfortunately, conditions of business generally were far from good in the early part of the year, the depression being common to all parts of the world and leading to serious monetary crises which are not, even now, past. Towards the latter part of the year, however, there has been a distinct revival of business in the dye world, accentuated by the fact that this country has gone off the Gold Standard. Due to this fact it has been possible to open up a very useful trade on the Continent where previously business had been extremely difficult and we ourselves have been finding orders and inquiries from abroad coming along in a steady stream. With the prospect of a stable Government assured for a few years ahead, we think that manufacturers generally can look forward with every confidence to a largely increased volume of business in the New Year.

Indian Drugs and Medicines

THE imports of drugs and medicines amounted in value to Rs.194 lakhs, which is a fall of 13 per cent. compared to the imports in the previous year. Germany sent more synthetic sulphur at lower prices and Switzerland and Italy also increased their supplies, while natural camphor from Japan came on a much smaller scale. The imports of quinine salts also declined to 107,000 lbs., but there was no import of cinchona bark. The value of proprietary and patent medicines showed some decline, the United Kingdom being the principal sufferer. There was also a decline in the imports of preparations of opium and morphia.

Japanese Agar Agar Production

TOTAL production of seaweeds in Japan during 1929 was placed by the Japanese Ministry of Agriculture and Forestry at 788,000,000 lb. Of this figure the production of "Tengusa" (Gelidium) used for making agar agar aggregated 10,472,000 lb. Other varieties are used in the production of foodstuffs and food flavouring.

Essential Oil Imports of United Kingdom

IMPORTS of essential oils into the United Kingdom during the first nine months of 1931 amounted to 2,788,300 pounds, as compared with 2,745,000 for the corresponding period of 1930. The total value of importations declined from approximately £790,000 in the 1930 period to £525,000 in 1931.

Indian Chemical Notes

(FROM OUR INDIAN CORRESPONDENT.)

Inorganic Chemistry

THE department of inorganic chemistry and physical chemistry is in charge of Dr. H. E. Watson, and considerable research work was in progress. The work on sulphuric acid manufacture has resulted in a process depending on a vanadium catalyst and capable of application to small industrial units. The cracking of wood tar supplied by the Mysore Iron Works, Bhadravati, has been found to increase the yield of carboxylic acid and further attention has been given to oil hydrogenation. A systematic study of methods for estimating free chlorine in sterilised water has been made, various materials of commercial interest have been analysed, and weights and measures were standardised for governments.

Organic Chemistry

IN the department of organic chemistry, many fixed oils have been further investigated as to composition and utility of hydrogenation, including several seed oils and the oils of sardine, shark liver and dogfish-liver; besides chrysalis oil and the oil of the cantharis insect, from which cantharidin has been isolated in commercially profitable yield. Various Indian weeds have been examined as raw material for oxalic acid manufacture, while other natural products investigated include essential oils, drugs and alkaloids. Subjects of more academic interest investigated in this section embrace researches on optical and structural isomerism, synthesis of amino acids, dyestuffs, compound of arsenic and antimony having the possibility of medicinal application, alkaloids and hetero-cyclic compounds.

Printing Ink Industry

THE Industries Department of the Government of Madras has succeeded in manufacturing printer's ink of a suitable type from lampblack. The product was tried by the "Hindu" a daily paper in Madras for three consecutive days with great satisfaction. An important problem in the matter was to devise a simple method for manufacturing lampblack, and after some consideration it was decided that the waste lubricating oil in garages and petroleum crude oil could be utilised for the purpose. Another question was how to arrange for the material to be suitably burnt so as to yield the maximum amount of carbon. Many points had to be considered in determining the lay-out of a suitable plant. An improved furnace has finally been evolved for regular production which has given satisfaction.

Imports of Dyes

NOTWITHSTANDING a decline in quantity from 19,000,000 lbs. in 1929-30 to 16,500,000 lbs. in 1930-31, the imports of coal tar dyes recorded an increase in value from Rs.197 lakhs to Rs.208 lakhs. Aniline dyes formed 77 per cent. of the total quantity as compared with 70 per cent. in the previous year, while alizarine dyes continued to recede both in quantity and value. The average declared value of alizarine dyes remained practically unchanged while that of aniline dyes advanced considerably. Imports of Germany advanced to some extent while those from the United Kingdom receded. The imports of synthetic indigo amounted to 10,000 cwts. the principal source of supply being Germany. The United States supplied less aniline dyes but more alizarines than in the previous year.

Tung Oil Developments

THE cultivation of Tung tree has now been earnestly taken up in Burma on a plantation scale, by a firm at Hsum Hsai in the Northern Shan States, which, it is understood, is backed by a large association of paint and varnish manufacturers in England. The trees already planted are showing excellent growth and there are no symptoms of disease. It is intended to put down 750 acres during the coming season and if the trees in the original experimental area flower and fruit satisfactorily, to go on extending the area. The experiment will be watched with much interest.

Cresylic Acid in Australia

ONE producer of cresylic acid in Australia puts out about 80,000 gallons a year. Imports during the fiscal period 1929-30 amounted to about 50,000 gallons. The chief consumption of cresylic acid is by manufacturers of insecticides and disinfectants.

Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.

The Endless Controversy

IN the *Glasgow Herald* recently there has been a controversy that revives all the heresies concerning the effects of the Dyestuffs Act on the textile industry. It has been suggested by the critics of the Act that it has cost, as one writer asserts in the case of his firm, "tens of thousands of pounds," and it is even asserted that the Act is responsible for the decline in the export of coloured yarn and piece goods. In fact, one would gather, if these stories could be accepted, that the condition of the textile trade of Lancashire to-day is solely due to the operation of the Act. How fallacious all this is is shown by the fact that the decline in the export of plain goods is greater than in the case of the coloured. What, in addition, these critics continue to overlook is the necessity that brought the Act into existence and the advantage of having a new home industry in operation and protection against exploitation by foreign rings that could hold the entire textile trade at ransom if we were again to become dependent on foreign supplies.

Two New Blacks

Imperial Chemical Industries continue to issue new colours in an unending stream. This month we have two new Duranol colours, three new Thionol colours, and two new acid colours.

Duranol Black E, it is claimed, has dyeing properties superior to any direct black for acetate silk at present available and it is of immediate interest to every dyer of this material. Its special feature is its remarkably good affinity; this enables it to build up readily and evenly and to exhaust well. When dyeing, matching is easy as the black appears as a black throughout. The dyeings illustrated show that in dyeing a 30 per cent. shade, under winch conditions, a black is obtained after two minutes. This colour can therefore be used in the jig, which is not possible with other available direct blacks. In the machine also the colour builds up quickly and readily.

Duranol Black G is suitable for application to all types of cellulose-acetate silk, and to mixed fabrics containing acetate silk. An important feature of this new dyestuff is that shades of increasing strength build up from pure greys to full blacks; a feature not usually found in blacks of this type. For this reason when using Duranol Black G there is less danger of "ending" troubles and in addition to its use in the production of full blacks Duranol Black G is of considerable value both as a shading colour and as a product for the dyeing of greys.

Thionol Colours

The three new Thionol colours, Dark Brown 3R, Dark Brown 4R, and Dark Cutch B, are recommended for the production of full, rich brown shades of a reddish tone on all forms of cotton and should be of special interest to yarn and loose cotton dyers for cross dyeing, washing and milling shades and to piece dyers for washing materials. They can be used in circulating machines for dyeing cops, beam warps, etc. The freshly dyed shades of these colours gradually become somewhat redder and brighter on exposure to the atmosphere. This final shade may be obtained by adding a little acetic acid to the last wash water after dyeing. When the shades are developed in this manner

they possess the advantage of being more stable to conditions of storage and acid atmospheres than many sulphur colours used for the production of similar shades.

An I.C.I. Speciality

Coomassie Brown GS is an I.C.I. speciality and possesses many novel and valuable properties. It is a homogeneous dyestuff which gives rich reddish brown shades when dyed on wool or silk from a neutral or mildly acid dyebath. It is of outstanding interest for dyeing loose wool and slubbing for the production of brown shades fast to washing, milling, perspiration, carbonising, stoving and decatizing. It may be dyed in the presence of chrome, the shades becoming yellower and somewhat deeper and improved in fastness to washing. It is of interest in piece dyeing for the production of medium to deep brown shades. To obtain the same standard of all-round fastness it would normally be necessary to use chrome dyestuffs.

This colour is extremely valuable for union dyeing. Its excellent affinity for wool or silk from a neutral dyebath makes it eminently suitable for all types of union dyeing. It is of the greatest interest to garment dyers taking into consideration the importance of brown shades. Coomassie Brown GS is eminently suitable for dyeing natural silk-cotton hosiery. It serves as a basis for numerous browns and fawn shades. The dyeing process is simplified by the very low degree of staining of the cotton which frequently causes difficulty in obtaining perfectly solid effects. The dyed shades are of good fastness to washing. This colour is of value for dyeing natural silk yarns and pieces. It possesses excellent affinity for both unweighted silk and tin-weighted silk and may be applied to the latter fibre from a neutral soap bath. It will be of value in printing as a ground shade on wool, silk or tin-weighted silk for the production of white and coloured discharges.

A New Blue Acid Dyestuff

Disulphide Blue EGS, an addition to the I.C.I. range of acid dyestuffs, is specially suitable for dyeing yarns and piece goods where brilliancy of shade combined with good fastness to washing is required. It is of value for use in combination with chrome dyestuffs, being very serviceable for brightening purposes. The shade is only slightly affected by the chrome, becoming somewhat yellower. It is suitable for dyeing the wool or the silk in union materials from an acid dyebath and leaves the cotton unstained. A point of interest in connection with piece dyeing is that this dyestuff does not stain cotton or artificial silk effect threads. Its brilliancy of shade makes it of value in silk dyeing, but it is not recommended for application to tin weighted silk. This colour possesses exceptionally good solubility and therefore is of special interest for printing wool and silk. It is suitable for lake making purposes for the production of lakes to be used in the printing ink and paper surfacing trades.

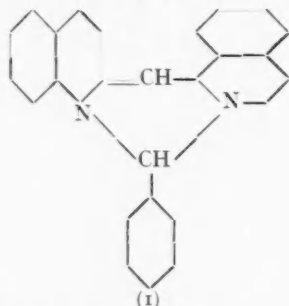
Additional Chlorazol Colours

Since the publication of Imperial Chemical Industries pattern card of Chlorazol Colours, eight new colours have been added to this well-known range, namely, Fast Orange ERS, Fast Orange WS, Purpurine 10 BS, Bordeaux 6BS, Catechine BS, Blue GS, Diazo Blue 2RS, Black FBWS.

Basic Intermediates for Dyestuffs: No. XLIV.—Quinoline and Related Compounds

By "Consultant"

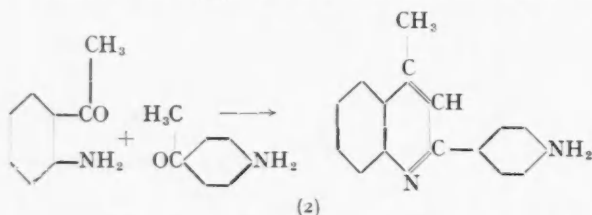
THE early days of the dyestuffs industry saw the introduction of a number of quinoline dyes, such as Quinoline Blue or "Cyanine," which were rapidly superseded owing to their fugitive nature, and, in some cases, to their high cost. Among them it is interesting to note the only derivative of *iso*-quinoline that has been applied to the dyeing industry, namely, *Iso*-quinoline Red. This was a most delightful shade of rose cerise, but its inability to withstand the rigours of exposure to light and laundering rapidly led to its obsolescence. It was made by heating the crude coal-tar quinoline, which contains both the normal and the *iso*-base, with benzotrichloride; in addition zinc chloride may be used to hasten the condensation, but this is not essential. The dye has the constitution shown in the formula (1) and has some interest to the chemist in view of



(1)

the fact that it led to a study of the photosensitive dyes, a matter which is more fully dealt with later in this article.

At the present time only two of the quinoline dyes are used for the purpose for which they were intended, namely, dyeing; the others are used in various applications of photography. Of the two dyes from this series, Quinoline Yellow, still finds a limited application for the production of fast, clear yellowish-green shades on silk or wool; the other, nearly obsolete, is Flavaniline, remarkable for its method of preparation. When acetanilide is heated with half its weight of zinc chloride to a temperature of 260° C, a series of reactions takes place which end in the formation of Flavaniline, α -aminophenyl-lepidine (2). This very peculiar reaction depends on the fact that the action of condensing agents on acetanilide is to form a mixture



(2)

of ortho and para aminoacetophenone. This is another instance of the well-known reaction of "para-migration" upon which the formation of benzidine and similar compounds rests. The aminoacetophenones then undergo a further condensation with the elimination of two molecules of water, leading to the production of a substituted lepidine derivative. This happens to have a deep yellow colour, but its use as a dye is limited by the fact that it is not very soluble; the action of "oleum" converts it into the monosulphonic acid, which is sufficiently soluble for use as a dyestuff. It is, however, much easier to produce the greenish yellow colour of Flavaniline by cheaper alternative methods, so that its manufacture has now stopped.

Quinoline

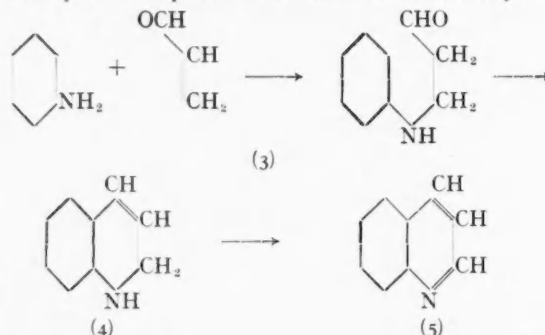
Although it is customary to refer to quinoline as a primary coal-tar intermediate, its extraction from this source is not a particularly easy matter, and most of the available commercial quinoline is synthetic. The quinoline of coal tar is accompanied by *iso*-quinoline, the isomeric α - and γ -methyl-quinolines (quinaldine and lepidine) and higher homologues.

The acid extraction of the heavy oil fraction from coal-tar yields a solution which on the addition of alkali gives an oil containing all these substances; if this be fractionated a so-called "coal-tar quinoline" is obtained, which has the following composition:—

	Amount.	Boiling point.
Quinoline	47 per cent.	239° C.
<i>iso</i> -Quinoline . .	20 per cent.	240.5° C.
Quinaldine . . .	25 per cent.	247° C.
Lepidine	8 per cent.	257° C.

The proximity of the boiling points of these substances makes their separation impossible by fractionation methods, and although there is a method of obtaining the *iso*-quinoline in a tolerable state of purity on account of the comparative insolubility of its acid sulphate, the quinoline cannot be so purified.

Synthetic quinoline is almost exclusively prepared by the method devised originally by Skraup, and familiar to every student of organic chemistry as a preparation of more than usually tempestuous character. The large scale method follows the laboratory method in many ways save that arsenic acid is preferred as the oxidising agent in place of nitrobenzene, since the reaction is more readily controllable. In practice the following is a typical method of manufacture. The vessel for the reaction consists of a resistant cast-iron pot fitted with clamped lid and appropriate glands for stirring gear, thermometer, a wide and very efficient reflux condenser; an internal cooling coil is also a necessity. The ingredients for the reaction mixture are as follows, aniline ($\frac{1}{2}$ cwt.), glycerine ($1\frac{1}{2}$ cwt.), arsenic oxide ($\frac{1}{2}$ cwt.) and sulphuric acid (98 per cent., $1\frac{1}{2}$ cwt.). These are thoroughly mixed by means of the stirring gear, and when thoroughly incorporated the steam is turned into the heating coils. At a certain temperature, bubbles will be seen coming from the body of the liquid; at this instant both stirring and steam are turned off and the reaction proceeds of its own accord; if more vapour is evolved than the condenser can cope with, cold water may be circulated through the cooling coils, but it is advisable to start the cooling a few minutes before it is absolutely necessary, or the reaction may go out of control. After the tumult has subsided, boiling for three hours is necessary to complete the condensation. To isolate the quinoline, the condenser is set for distillation and 15 per cent. sodium hydroxide is run into the kettle. When alkaline, steam is blown through and a mixture of aniline and quinoline distils with the water. These can be separated with a good column, or the older method of separation may be adopted, namely, to cool the solution of the mixed bases in dilute sulphuric acid and run in sodium nitrite until diazotisation of the aniline is complete. On gently warming this solution with direct steam the diazonium base is converted to phenol; the liquid is then made alkaline and the quinoline steamed out. Actually this chemical separation is seldom called for. The chemical mechanism for the conversion of aniline to quinoline depends on the formation of acrolein by the



(3)



(4)

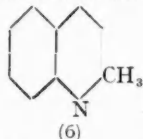
(5)

oxidation of glycerol, but the reaction between this aldehyde and aniline is complex. There is undoubtedly some of the Schiff base formed by the loss of the elements of water from the

oxygen of the -CHO group and the hydrogen of the -NH₂ group, but the main reaction is probably the addition of the amino group across the unsaturated link of acrolein to give the compound shown in (3). Ring formation then takes place with loss of water leading to a dihydroquinoline, which is readily oxidised to quinoline itself (5). As prepared commercially quinoline is an oily colourless or pale brown liquid with a strong, but not too unpleasant smell.

Quinaldine

The manufacture of quinaldine follows out a chemical procedure which is analogous to that used in the preparation of



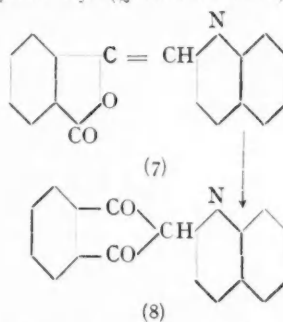
quinoline itself. The aldehyde is not, however, prepared *in situ* as in the case of quinoline, but is obtained as a separate stage and is condensed with the aniline according to the method of Ullmann. The aldehyde in question is acetaldehyde.

To prepare quinaldine or α -methylquinoline (6), aniline hydrochloride ($\frac{1}{2}$ cwt.) is vigorously stirred with acetaldehyde (44 lb.) and water (8 gall.) for several hours. The reaction that takes place under these conditions is exothermic and the temperature must be kept below 30° C. by the use of internal cooling coils. After six hours' stirring in this way the cold water is changed for a mere trickle of steam—just enough to keep the temperature up to 25° C. at which point it is maintained for 64 hours. The end of the condensation can be ascertained by withdrawing a small sample and warming with sufficient caustic soda solution to give an alkaline liquid. If any appreciable amount of aniline hydrochloride remains, an oil separates; on the other hand, if the condensation is complete, a crystalline base separates in white flocks. At this point the material is transferred to a still, diluted with twice its bulk of water, made strongly alkaline with caustic soda and the quinaldine distilled off in steam. During this distillation the

contents of the still must be vigorously stirred to prevent caking of the base, and loss of quinaldine. The yield from the above process is dried and fractioned, the fraction from 230—270° being collected. It should weigh 22–24 lb.

Quinoline Colours and Photosensitising Dyes

The only quinoline dye now prepared in bulk is that obtained by heating phthalic anhydride and quinaldine in the presence of some condensing agent such as zinc chloride. The substance produced has the constitution shown in (7), but it rapidly turns into Quinoline Yellow Base (8). This substance when treated with oleum gives a disulphonic acid which is used as a greenish yellow dye (Quinoline Yellow).



Well over half a century ago, in 1873, Vogel found that ordinary photographic plates—insensitive to red light in the normal course of events, became sensitive to this form of light after impregnation with certain dyes. Of these Isoquinoline Red proved to be the most valuable of the then known dyestuffs for this purpose, and plates treated with it had a better colour balance than any hitherto prepared. This led to a wide investigation of the whole series of Quinoline dyes by Pope, Mills, Hamer and others, and incidentally, to the introduction of panchromatic plates into standard photographic practice. The dyes of this group will be discussed in the next contribution to this series.

Safety in Artificial Silk Factories

By Dr. J. A. Lloyd

The following paper was presented at the Chemical Session of the National Safety First Week at Leeds, May 11–16, by Dr. J. A. Lloyd (of Courtaulds).

In the artificial silk factories at Courtaulds, we observe as strictly as possible all the Model Safety Rules such as are embodied in the booklet issued by the Association of British Chemical Manufacturers, and in particular those indicated by the lecturer with regard to the cleaning and repairing of tanks and other vessels. In addition, each factory has its Safety First Committee consisting of three responsible men, who at intervals make a definite tour of inspection all over the factory, and write down their recommendations in a special book kept for the purpose. These recommendations have to be carried out. Not even the managing director cancels them.

Cleaning and Repairing Tanks

With regard to the cleaning and repairing of tanks and other vessels, we appear to have had few, if any, serious accidents in connection with the sulphuric acid or the carbon bisulphide storage tanks. On the rather rare occasions that a man has to enter a sulphuric acid storage tank, the tank is, before entering, thoroughly washed out and filled with soda solution to neutralise the last traces of acid. With regard to carbon bisulphide tanks, the arrangements are very simple. No man is under any circumstances whatever allowed to enter a carbon bisulphide tank. When cleaning out is necessary, the carbon bisulphide is displaced by water, the manhole covers are removed, the water partially drained off, and the bottom of the tank freed from deposit by puddling. The sludge is then pumped out or ladled out with brass scoops, avoiding the use of iron, which might give a spark. It is of great importance that the tanks should not be allowed to become dry, because there is invariably a deposit of sulphide of iron in a very finely divided state, and this is liable to

spontaneous combustion on becoming dry, giving rise to fires and even explosions. If a carbon bisulphide tank is worn out and requires scrapping, it is half-filled with water, after which two or three of the plates are cut out from the top half; the water is then pumped out and the shell left exposed to the air for several weeks.

With regard to storage vats such as are used for dilute acid, alkali or other liquids, either covered or not, no man is allowed to enter without previous permission in writing from the chemist or engineer in charge, and he is compelled to wear a life-line held by another man outside the tank. Further, if there is danger of noxious gases being present, a gas mask is also insisted upon.

Fatal Accidents

Although Courtaulds have been fairly free from serious accident, there have, unfortunately, in past years occurred two or three fatal accidents. A few years ago, in a factory on the other side of the world, a man was working in a large lead-lined vat, which had not been properly isolated from the other vats and pipes through which liquid was passing, with the result that the gases given off by this liquid entered the vat, and gassed the workman before he could escape. This severe lesson has, of course, taught us to blank off, or otherwise securely close, every pipe leading to a vessel in which a man is about to work.

The Effluent Problem

Another feature of viscose artificial silk factories is the effluent. In point of fact, there are two or three different kinds of effluents. One contains dilute sulphuric acid, another contains sodium sulphide, and there is often a third consisting of caustic soda solution more or less carbonated

We have found it particularly important that these effluents should be conveyed to the effluent works in separate pipes, so that no opportunity is given for them to mix before they reach the effluent works. Some nine or ten years ago, some sulphuric acid effluent and the carbonated caustic soda effluent became accidentally mixed, passing through one of the cellars. Several men entered the cellar. One sat down to change his clogs and remained in this position while all his mates simply walked through the cellar and out at the other end. The one poor man, however, who sat down got his head into the belt of carbonic acid, and before it was noticed that he was remaining still for too long, asphyxiation had taken place. Since then, the effluent pipes have been kept strictly apart. There is another point I would like to raise. We find that many of the workmen do not realise sufficiently the dangers that exist, nor do they fully appreciate the rules which are made to safeguard them, so that it has been found to be specially necessary to fix large notices, and insist on them being followed, under penalty of instant dismissal.

It is perhaps scarcely worth while speaking of an episode of a considerable number of years back, which would not be able to take place under the present conditions insisting on the use of life-lines. The hero related his experience to me a few days ago. On opening a manhole where two drains met, he noticed that the outlet was becoming choked by a board which had got lodged there. He immediately took off his coat and climbed down through the manhole, but no sooner was his head inside than he collapsed and fell into the water. His mates, however, were fortunate in being able to pull him out with a boat-hook, but he is a broadly built man, and they had some trouble, and he assures me that he still bears two scars from that boat-hook. However, all's well that ends well, and he went home and slept for twenty-four hours on end, and then, in his own words, he got up and ate the biggest breakfast he ever had in his life! That happened many years ago. He learned his lesson, however, and has never since allowed any man working under him to enter a manhole or tank without a life-line.

Water for the Textile Industries

By A. S. Behrman

The following notes on water for use in the textile industries are taken from a recent address by Mr. A. S. Behrman, chemical director of the International Filter Co., Chicago.

WHAT are the qualifications of a good water for textile use? The water must be clear—that is, it must be free from turbidity or suspended matter of any kind, in order to avoid muddying and spotting the fabric, and to prevent uneven dyeing. The water must be colourless, or it may impart some of its colour to the fabric. The water must be soft, in order to permit thorough and economical washing, and in order to avoid spotting, uneven dyeing, and the host of other evils which are too well known to need further comment. The water should be free from iron, to prevent rust stains and other troublesome precipitations. In rayon work in particular, the complete absence of iron is of prime importance. Finally, the water should be non-corrosive, since even if initially pure, it may become thoroughly undesirable if it attacks iron or other metallic surfaces with which it comes in contact and so is contaminated with compounds of the metal.

Clear Water

The practically universal method of removing mud or other suspended matter from water is by means of filtration through sand. It is not enough, however, to rely on the sand alone; for a sand bed, like any other strainer, can only remove particles of suspended matter which are larger than the spaces between the sand grains. Since these spaces are a great many times larger than the tiny particles of silt, clay or other similar causes of turbidity, it is obvious that passing a turbid water of this sort through a sand filter at the high rates now employed would result simply in straining out the coarsest particles of suspended matter, while the finer particles would go through.

This situation is met by adding to the water one or more coagulants—such as alum, sulphate of iron, sodium aluminate, ferric chloride, etc.—whose function is to collect these very fine particles of suspended matter into clusters or clumps which are larger than the spaces between the sand grains, and which will, therefore, remain on top of the filter sand when the water is passed down through the bed. All of these coagulants form in water a jelly-like substance which attracts the tiny particles of suspended matter and gathers them into the desired larger aggregates.

In some cases it is sufficient to add the coagulant to the water just as it enters the sand filter. This is the case in the usual pressure filter installation. Frequently, however, additional time must be provided for the coagulant to do its work before the water passes to the filter.

Colourless Water

The colouring matter of natural waters, is, for the most part at least, not really in solution at all, but in the form of a very fine suspension. A water may be coloured a bright yellow or light brown and look perfectly clear to the casual observer; but it may readily be shown that the particles of colouring matter are in suspension by treating the water with alum or some other coagulant, whereupon the coloured particles are brought together, and can then be removed by settling or filtration, or both.

It is general practice to remove colour from natural waters by coagulation and filtration, so that in many cases of surface waters which are both coloured and turbid, the colour and turbidity are removed at the same time. It is usually, however, a much simpler job to produce a clear water than a colourless water, and it is here that the chemist becomes especially important in working out the most effective and economical method by which the colour may be removed.

In some waters the colour can be bleached out with chlorine. This fact has suggested a new method of colour removal which consists in destroying the colour with an excess of chlorine and then removing the excess of chlorine with activated carbon. Hydrosorco, the activated carbon used by the International Filter Co., has a remarkable affinity for chlorine, and combines chemically with it. Where it is possible to remove the colour from a clear water by means of excess chlorination and subsequent treatment with activated carbon, coagulation and sand filtration may be eliminated entirely.

Soft Water

Few industries have realised the importance of soft water as quickly as the textile industries. It is not enough that the water must be reasonably soft; for many textile purposes the water must be entirely free from hardness. For this reason the textile industries were among the very first purchasers of zeolite softening plants, and now it is only the exceptional textile works which is not thus equipped.

We have become so accustomed to our zeolite softeners that we are very likely to forget that no one—not even chemists who are charged with developing “bigger and better” zeolites—knows exactly how a particle of zeolite operates in exchanging its soda for lime and magnesia of the hard water with which it comes in contact. We have our theories and our suspicions, but up to date there is no generally accepted picture of just what takes place in a zeolite molecule in either the softening or regeneration procedures.

Non-Corrosive Water

The last few years have seen a decided advance in our understanding of why some waters will corrode iron pipe and some will not. The corrosion of iron pipe by water may be determined pretty definitely by an analysis of the water. Provision should, therefore, be made in advance either to treat the water so that it will be non-corrosive, or to make the piping and other metal surfaces with which the water comes in contact of materials that are in themselves non-corrosive.

Of steadily increasing importance is the purification of waste liquors from textile works, and particularly from dye houses. Up to the present, the subject has been of practical importance in only those few states in which the health boards have police powers and can compel the owner of an industry to stop polluting a water course. In the rest of the states, in the absence of such police powers in the health boards, most of the industries are trying to put off the evil day as long as possible and are content to let matters take their course.

With our steadily growing population, and the ever-increasing use of surface water for public water supplies, it is only reasonable to assume that the day of reckoning is not far off, and is approaching with accelerated rapidity. It would, therefore, seem to be wisdom on the part of the textile industries to be prepared for the day of judgment, and possibly even to anticipate it, since a programme voluntarily adopted after an intelligent analysis of the situation may well be considerably more effective and economical than one imposed by governmental regulation.

There are so many variables which may enter into the composition of waste liquors that the most advantageous method of purifying them can be diagnosed accurately only after a careful and complete analysis of conditions—not only as to the character of the liquors themselves, but as to the operation of the plant, the character of the adjacent drainage area, and other considerations which are peculiar to the particular case at hand. Here again the chemist and the chemical engineer must play an important part in working out the most economical and effective treatment.

Dyes and Their Application: Recent Technical Progress

By L. J. Hooley

Cellulose Acetate Dyeing and Processing

METHODS of dyeing and printing and new dyes for cellulose acetate as well as processes which do not involve colouring such as delustring are still appearing in sufficient volume in the literature, especially the patent literature, to show that this remains one of the important research fields in colour and textile chemistry. They are chiefly concerned with the removal of secondary difficulties in the use of this special fibre, the primary ones having been already overcome.

Dyeing Processes

In B.P. 313,450 (British Celanese, Ltd.), the process of B.P. 282,036, of dyeing from solutions in organic solvents with fugitive colours is extended to dyeing with permanent colouring matters by

- (a) dyeing similar threads while they are travelling,
- (b) by printing, spraying and stencilling methods.

Solvents which evaporate slowly such as polyhydric alcohols, their ethers and esters, and ethylene, diethylene and propylene glycols are cited as suitable.

Hydrogenated isocyclic bases form useful assistants in dyeing as they are weakly alkaline and facilitate the dyeing of cellulose acetate without being sufficiently powerful to cause any harmful action to the material. Hexahydromethylaniline, hexahydroethylaniline, hexahydrotoluidine and hydrogenated naphthalenediamines are mentioned as suitable for the purpose. Where an acid dyestuff is employed a salt of the hydrogenated isocyclic base is substituted. An example is given of dyeing with the sodium salt of 1-amino-4-hexahydroanilidoanthraquinone-2-sulphonic acid in a 1 per cent. solution of hexahydroaniline (B.P. 293,766, I.G.F.A.-G.). This process is extended in B.P. 320,422.

R. Metzger recommends sulphonated mineral oils for dyeing acetate silk with colours of the insoluble type. (U.S.P. 1,738,660.)

For the dyeing of mixed fabrics containing acetate silk, preparations incorporating diazotisable bases with a wetting agent consisting of a naphthalene sulphonic acid substituted by a hydrocarbon residue, are convenient as the resulting products are soluble in water and after absorption by the acetate silk can be diazotised and developed. (B.P. 299,787, I.G.F.A.-G.)

In using sulphur colours for dyeing mixed fabrics, while this has been carried out from baths containing soap or substances such as sulphite cellulose waste liquor, the method is stated to be liable to cause staining of the acetate silk and it is claimed that for this reason it is better to dye from a bath containing the sodium sulphide without any added matter or even more satisfactory to replace the sulphide with a less alkaline reducing agent such as hydrosulphite, at the same time adding a salt such as borax or sodium bicarbonate with a weak acid character. Where a two-colour effect is required the acetate silk can afterwards be dyed with a colour which will not affect the cotton. (B.P. 320,027, I.G.F.A.-G.)

Another variation of the amino oxidation method for blacks consists in impregnation from an aqueous solution or suspension of aniline containing up to about 10 per cent. of *p*-aminodiphenylamine or *p*-phenylenediamine, and then developing on the fibre by hot acid dichromate, preferably with the addition of a small quantity of an oxidising agent such as copper sulphate. (B.P. 337,746, A. J. Hall.)

For dyeing mono- and di-acetylated celluloses, which are obtained by the direct acetylation of fibres without structural alteration in processes of the immunisation type, it is con-

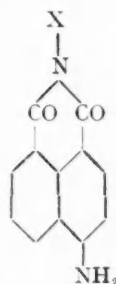
venient to carry out the dyeing while the fibres are still in the swollen state resulting from the acetylation process. (B.P. 323,011, Sandoz.)

In the film form, cellulose may be coloured by vat colours by treating with solutions of leuco compounds with the addition of a small quantity of an alkyl naphthalene sulphonic alkali metal salt as sole wetting agent, and then converting the leuco compounds into the dyestuffs within the fibre. As an example, Cellophane is slowly passed through a bath containing Anthra Red B, caustic soda, sodium hydrosulphite and isopropyl naphthalene sulphonate of soda, the rate being adjusted so that each portion of the film is in contact for about 10 minutes. After freeing from excess liquor by passing through two rubber stripping flaps the film is submitted to oxidation in the air for one minute, then washed with water to remove alkalies and salts and dried. (B.P. 320,055, I.G.F.A.-G.)

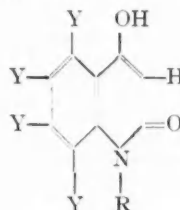
New Dyes

Derivatives containing the tetrahydro-naphthalene nucleus are used in the dyeing of acetyl silk in B.P. 330,591 and B.P. 330,602 (British Celanese, Ltd., H. C. Olpin). In the former an azo dye such as tetrahydro- β -naphthylamine coupled with phenol is used, the products being in the neighbourhood of yellow and orange in shade, while in the latter imide derivatives obtained by condensing 1-chloro-2:4-dinitrobenzene or a similar nitro compound with a reactive halogen atom with *av*-tetrahydronaphthylamines are used. The products described are yellow and generally speaking a sulphonic acid group is undesirable.

The I.G.F.A.-G. use 4-amino-1:8-naphthal-hydrazide for obtaining greenish yellow dyes for acetyl silk (B.P. 338,085). They also use a somewhat similar series of compounds in B.P. 328,308 where deep yellow tints with a greenish hue are obtained from derivatives of 4-amino-1:8-naphthalic acid of the general formula, where X stands for a cyclohexyl residue

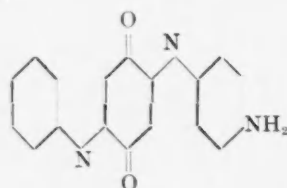


In B.P. 327,394 (I.G.F.A.-G.) quinolone derivatives corresponding to the general formula



in which R stands for an alkyl radicle which may be substituted and Y for hydrogen and any appropriate substituent, are coupled with diazo compounds, both these and the quinolones being free from sulphonic and carboxylic acid groups. Thus the product prepared by coupling diazotised m-nitraniline with N-methyl-4-hydroxy-2-quinolone dyes acetyl silk in very fast green yellow shades.

Among the derivatives of quinones, other than anthraquinone, which have been suggested for dyeing, there are the N-arylated nitrogen derivatives of benzo- and naphthoquinones and also some new products obtained from naphthazarine. These latter are made by treating naphthazarine with ammonia, by an analogous process to that which has been used for obtaining amino-anthraquinones from leucoquinizarine. The body obtained by treating naphthazarine with ammonia and sodium hydrosulphite in aqueous solution and then oxidising to reconvert the leuco body to the quinone gives grey shades. The N-arylated quinones may be illustrated by 2:5-di-p-amino-phenylamino-benzoquinone (obtainable by the action of p-phenyldiamine on benzoquinone) which gives brown shades (B.P. 322,463, and 327,860. Brit. Celanese, Ltd.).



To the list of azo dyes which have now been specified there may be added the carboxylated azo dyes, other than derivatives of salicylic acids, such as, for example, p-nitroaniline → 4-hydroxy-α-naphthoic acid (R. Metzger, G.P. 457,957) and azo dyes obtained by coupling one, two, three, or more molecular parts of the same or different coupling compounds with diazotised amino-nitro compounds containing two or more aryl residues (B.P. 323,792. Brit. Celanese, Ltd.).

Delustring

Owing to its greater similarity to natural silk in appearance delustred silk is becoming of increasing importance. Recently A. K. Johnson (*Text. World*, 1930, 1388) has classified the various methods of carrying out delustring as follows:

- (1) Processes involving alteration in the substance of the fibre or distribution at the surface.
- (2) Deposition of foreign matter on the surface.
- (3) Physical abrasion by chalk, etc.
- (4) Alterations in filament position by varying the twist in this yarn or allowing some shrinkage in finishing.

Some of the methods mentioned more recently are given below.

In U.S.P. 1,756,941 (C. Dreyfus, H. Platt) a zinc salt is impregnated in the material in the presence of a swelling agent and the material, after standing for some time, then treated with a sulphide, so that zinc sulphide is deposited within the fibre. This zinc sulphide also improves the affinity of the material for direct dyestuffs. A rather different method is that of B.P. 333,504 (Brit. Celanese, Ltd. W. I. Taylor) in which a wetting agent is incorporated with an organic solution of the cellulose acetate and this is allowed to set under such conditions that it remains in the silk. In B.P. 335,204 (Brit. Celanese, Ltd., W. I. Taylor, C. Dreyfus) the bases previously applied to threads are used with woven and knitted fabrics. In B.P. 313,072 (Soc. Pour de la Soie "Rhodiaseta") the material is treated with a substance such as cyclohexanone, ethyl benzoate or anisole, which has a swelling action but which is insoluble in water. The use of a high temperature is not essential and 40° C. may be found sufficient, although as high as 80° may be used.

An opposite process to those above is illustrated in B.P. 306,534 (Brit. Celanese, Ltd.) where an agent adapted to prevent delustring or to restore the lustre where it has been removed is used in printing, the agent in question being added to the printing paste and consisting of a substance such as the methyl or ethyl ethers of ethylene glycol, with or without further substances like alcohol, benzyl alcohol, cyclohexanone.

Scottish Dyestuffs Report

ALTHOUGH conditions in Scotland in general have not shown much change, the month has ended on a more optimistic note. How far this is due to actual direct improvement in orders and how far to the improvement in the world outlook following the Hoover proposals and the resulting rises in the stock market, it is, of course, difficult to say. Possibly also the approach of the holidays is having a psychological effect. It was indicative of the state of trade that advance bookings at the usual Scottish holiday resorts had been disappointing, but with the beginning of July reports are that last-minute arrangements are now bringing numbers to nearer normal.

Dye sales have been fairly good, and vat dyes have now been firm for some time. Dyers in the Glasgow and other districts have not been too well off, and printers have, of course, been hit by recent closings down. Chemical sales have been steady.

Under-production is still too evident in the South of Scotland woollen industry, and a number of manufacturers have only about half their looms running; orders from abroad have, however, helped the situation, and the spring season's orders have picked up a little.

Dr. B. Wylam, who has been a research chemist, first with Morton Sundour Fabrics and then with Scottish Dyes during the last eight years, has recently left to take up his new appointment of Inspector of Factories for Scotland, in place of Mr. J. W. Young, who has retired.

Dyestuff Licences for June

THE following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during June has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee:—

The total number of applications received during the month was 596, of which 491 were from merchants or importers. To these should be added 11 cases outstanding on May 31, making a total for the month of 607. These were dealt with as follows:—Granted, 556 (of which 515 were dealt with within seven days of receipt); referred to British makers of similar products, 45 (of which 39 were dealt with within seven days of receipt); outstanding on June 30, 6. Of the total of 607 applications received, 554, or 91 per cent., were dealt with within seven days of receipt.

Delustring Cellulose Acetate Rayon

THE interesting discovery has now been made (E.P. 343,121) that whereas cellulose acetate (or other cellulose ester and ether) rayon has no affinity for inorganic substances when applied from simple aqueous solutions, this rayon readily absorbs these substances from their colloidal solution. The discovery is usefully applied for purposes of weighting or reducing the lustre of cellulose acetate rayon.

The oxides of aluminium, titanium, silicon, zinc, and cerium, barium carbonate and sulphate, and zinc sulphide, are among the substances which can be absorbed by cellulose acetate rayon when immersed in their colloidal solutions. Bentonite, a finely divided naturally occurring silicate which is also used for softening textile materials. If cellulose acetate rayon is treated with a solution consisting of:

Bentonite	400 parts
Methylated spirit	100 "
50 per cent. gum Arabic solution	50 "
Phenol	25 "
Water	425 "

absorption of the bentonite occurs such that the rayon loses much of its lustre; it then resembles real silk more closely.

Zinc sulphide appears to be an excellent delustring agent for cellulose acetate rayon and E.P. 344,093 describes methods for utilising this substance. The most satisfactory method consists of precipitating the sulphide within the rayon by double decomposition between a zinc salt and sodium or other sulphide. In order to facilitate the absorption of zinc salt solution, the rayon must first be swelled, or alternatively the swelling and absorption may proceed simultaneously. Thus in a satisfactory delustring process the rayon is immersed padded with a solution of zinc chloride (250 parts of zinc chloride, 100 parts of formic acid, and 750 parts of water) and then treated with a solution of sodium sulphide.

Dyestuffs Monthly Supplement

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Colour Users' Policy

THE annual address by Sir Sutcliffe Smith, the chairman of the Colour Users Association, has been already noted. In the annual report of the Association emphasis is placed on the urgent necessity of keeping the Association in being in view of the situation likely to arise when the extended Dyestuffs Act expires on December 31 next. It is considered to be of vital importance that the Association should then be in a position to place the views of colour users before the proper authorities. There seems to be no better prospect of common agreement among all parties than there was during the autumn of last year, when several meetings were held without any definite result being reached. This year the Government have pronounced against any further inquiry and report, and the position, therefore, remains quite indefinite. If the present Government remain in office, it seems probable that the Act will not be renewed. A change of Government on the other hand might bring about a change of attitude.

Tributes to the Makers

There was no pleasanter feature in Sir Sutcliffe Smith's address than his tribute to the progress that British dyestuffs production has made in recent years and to the marked improvement in the technical services. It used to be the fashion to compare British and German services, very much to the disadvantage of the former, but now apparently there is little to choose between them. The large ranges of new colours now being produced here is certainly a matter for recognition, for only those in the industry appreciate the efforts and the expense involved in the production of a single new dyestuff. While all the makers are deserving of tribute, Sir Sutcliffe Smith particularly mentioned the large number of new colours produced by Imperial Chemical Industries, which of course means by the old British Dyestuffs Corporation and Scottish Dyes, Ltd., our own columns have borne testimony to the fact nearly every month. This month Imperial Chemical Industries announces the marketing of five new colours which are described in detail below.

Two New Acid Colours

Lissamine Blue BFS is an addition to the I.C.I. range of acid dyestuffs. It is eminently suitable as a basis for dyeing navy blue shades on all types of woollen and worsted piece goods. It is level dyeing and possesses very good fastness to water, washing, carbonising, stoving, decatizing and hot pressing. In addition, it does not stain either cotton or viscose effect threads. Suitable shading colours for use in combination with Lissamine Blue BFS are:—Lissamine Violet AVS, Lissamine Violet 2RS, Lissamine Green VS and Naphthalene Fast Orange 2GS. The washing fastness of this colour makes it of interest for dyeing wool yarns in medium and full shades. Lissamine Blue BFS is of interest in union dyeing where it is desired to dye the wool or silk in union materials, from an acid dyebath, the cotton to be left unstained. This colour is suitable for dyeing natural silk from an acid dyebath giving bright blue shades of fairly good fastness. Owing to its excellent penetrative power it is suitable for dyeing wool and fur felt-hat bodies. It is of interest for the direct printing of wool and carpet yarns, silk and tin-weighted

silk. Lissamine Blue BFS possesses good affinity for the wool fibre when dyed from a sulphuric acid dyebath. A feature of the colour is that it is absorbed by the fibre at a uniform rate and during the dyeing process it shows no stage where the rate of exhaustion is irregular. The colour is mainly used in medium and full shades, under which conditions it may be considered very level dyeing. It is, however, not suitable for shading purposes or for very light shades where small quantities are required.

Naphthalene Leather Brown OHS, another new I.C.I. acid colour, is a homogeneous product applicable to vegetable, semi and full chrome tannages, and possesses moderate fastness to light and good penetrative properties. The dyestuff is of excellent solubility and is therefore very suitable for brush stained work. A wide range of brown shades may be produced with Naphthalene Leather Brown OHS in combination with Naphthalene Black 12BS and Citronine RS. The method of application is that in general use for the dyeing of acid colours. Additions to the dyebath of formic acid or sulphuric acid are best made after the leather has been dyeing for 15–20 minutes. A further working for 20 minutes completes the dyeing operation. For brush staining this colour gives very level shades and may be used at concentrations up to 2½ ounces per gallon of water.

A New Caledon Blue

Caledon Dark Blue 3BS Paste, a further addition to the rapidly extending range of Caledon Colours, is suitable for the production of navy blues in all classes of cotton goods either in the open beck or in machines and is also adapted for the dyeing of viscose, linen and silk. As in the case of Caledon Dark Blue WFS, a special feature of the 3BS brand is the fastness of the dyed shade to moisture, the lack of which made matching-off a matter of considerable difficulty when using the older brands of vat dark blues.

A New Basic Colour

Victoria Pure Blue BOS, a new I.C.I. basic colour, gives blue shades appreciably brighter than those obtained from Victoria Blue BS and greener than those from Victoria Blue RS. Its chief features are a much brighter shade than Victoria Blue BS and much better solubility than Victoria Blue RS. It is recommended for application to all types of textile materials and may be used for all purposes where basic colours come into consideration and where brightness of shade is of primary importance. In application and general properties it may be regarded as being very similar to Victoria Blue BS and RX but possessing the above mentioned advantages of shade and solubility respectively. Particularly bright shades are obtained on natural silk and tin-weighted silk, and it is also very suitable for dyeing jute, coir, woodchip and allied fibres. It is of interest for dyeing wool where fastness to milling and stoving is of importance, as in blanket headings, etc. This colour is specially suitable for the production of lakes for use in the printing ink and paper surfacing trades, where brightness of shade is of great importance. It is eminently suitable for direct printing of cotton, viscose, wool, silk, tin-weighted silk and acetate silk.

The fifth new colour is Chlorazol Bordeaux 6BS, a addition to the I.C.I. range of Chlorazol dyestuffs.

Developments of the Society of Chemical Industry in Basle

By Dr. G. Engi

We give below extracts from an address delivered by Dr. G. Engi, Ph.D., D.Sc., at Salters' Hall, St. Swithin's Lane, London, on Tuesday, July 14.

It is not appropriate here to enlarge in detail on the position of the Swiss chemical industry, particularly of the Swiss dyestuff industry, in the compass of world economy. But it must, nevertheless, be emphasised in this connection that the natural pre-suppositions for a flourishing chemical industry, with some few exceptions, are not very favourable in Switzerland. Upon our country, lacking in raw materials, with its highly limited home market, its high wages, and social burdens, its high freights, a considerable restriction in the selection of the fields of activity is imposed. If the Swiss dyestuff industry, in spite of the unfavourable local and manufacturing conditions, has experienced a successful development, this is mainly due to its fertile inventive activity, and to the steadily pursued expansion of its basis of production, but also to the systematic improvement of the manufacturing processes and the apparatus and mechanical equipment, and to a suitable rationalisation kept within reasonable bounds.

The Indigoid Vat Dyestuffs

My inventions, dating back some years, in the field of the indigoid dyestuffs, have led to a range of vat dyestuffs comprising all the shades of the spectrum, reference being made especially to the condensation of oxythionaphthene or oxythionaphthencarboxylic acid with α -isatin arylides and other isatin derivatives substituted in the α -position, resulting in the important 2-thionaphthene-2-indoleindigo and the numerous substitution products thereof. The introduction of halogen into the molecule of indigoid dyestuffs, *inter alia* has also led to the important tribromo-, dibromo-, and monobromo-derivatives of the 2-thionaphthene-2-indoleindigo (Ciba Violet B, 3B, and Ciba Grey G), and to the polyhalogeno-indigos, especially the important tetrabromoindigo (Ciba Blue 2B).

The results of further research work in the field of the indigoid dyestuffs, include Ciba Red B, G, R, Ciba Pink B, BG, Ciba Bordeaux B, Ciba Orange G, Ciba Green G, Ciba Heliotrope B, Ciba Brown R, and the action of acid chlorides on indigo (Indigo Yellow 3G Ciba, Ciba Yellow G, Lake Red Ciba B).

This series of indigoid vat dyestuffs has of late been advantageously augmented in several directions by various thionindigo dyestuffs of the naphthalene series, to which belong, for example, Ciba Brown G and 2R, Ciba Violet 6R, Ciba Bordeaux G; further by the brands Ciba Orange R, Ciba Red 3B, various Ciba Brilliant Pink brands, and a series of stable solid and liquid leuco-vat preparations for wool.

Dealing for the present with the vat dyestuffs, I may instance the production of a complete range of vat dyestuffs of the anthraquinone series, which has advantageously become known under the name of Cibanone dyestuffs of our firm. In this connection I will merely make brief mention of two new classes of anthraquinone dyestuffs which have in more recent times been made accessible by the research work of our firm, and particularly have evinced also a considerable scientific interest. In 1921 and the following years a new series of patented vat dyestuffs of the anthraquinone series was produced by introducing the cyanuric ring into the anthraquinone molecule. This series is suitable for the dyeing and printing of cotton and is distinguished by great beauty and fastness properties. The most advantageous technical method for the production of these dyestuffs consists in the action of cyanuric chloride on aminoanthraquinone derivatives such as, for example, α - and β -aminoanthraquinone and the substitution products thereof, the aminoanthraquinone-acridones, the aminoanthraquinonexanthones, the aminoanthraquinone thioxanthones, and so forth. Other interesting substances are obtained by methodically causing cyanuric chloride and suitable acid chlorides, such as benzoyl chloride, to react on these aminoanthraquinones. In this manner yellow, orange, red, brown, green, and violet dyestuffs are obtained.

Indigoid and Thioindigoid Derivatives

Particular scientific interest was also attached to a further series of vat dyestuffs which was produced and patented in 1923, and with the manufacture of which the already long-

sought synthesis of indigoid and thioindigoid derivatives of the anthracene or anthraquinone series was realised for the first time. They were produced by making use of the experiences gained previously in the manufacture of indigoid dyestuffs, by converting the 2-thioglycol-3-carboxylic acid of anthraquinone and anthracene into their oxythionaphthene derivatives, and condensing the latter with a large variety of substances, such as, for example, isatin or its α -substitution products, asenaphthenequinone, and some others. According to these processes a series of indigoid and thioindigoid dyestuffs of the anthraquinone and anthracene series of a variety of shades and in general satisfactory properties of fastness were produced.

Some years ago we undertook the manufacture of a series of blue-dyeing vat dyestuffs from carbazole-indophenols according to patented processes. These are on the market as Ciba Blue 2RH, RH, and BH.

Chromium Compounds of Azo Dyestuffs

A number of years ago an observation, which proved to be of considerable importance, was made, namely, that certain chromium compounds of azo-dyestuffs containing groups capable of being chromed, when using a suitable dyeing process, are excellently adapted for the dyeing of animal fibres after the manner of the acid dyestuffs. Chromium compounds of azo-dyestuffs containing groups capable of being chromed had shortly before been obtained by R. Bohn by treating certain *o*-oxyazo-dyestuffs with chromic salts in presence of acid-binding agents, but had only been used for the production of inks and for printing vegetable fibres. Our Society has subsequently found and patented a series of new processes for the production of new complex chromium compounds of azo-dyestuffs containing chromable groups. By a suitable selection of the starting materials, and the use of particular dyeing processes, these products can be fixed on animal fibres, producing shades of great beauty and fastness. The first Neolan dyestuffs originated in this manner.

By applying these new processes to certain *o*-oxyazo-dyestuffs, which as after-chroming dyestuffs possess no special value, since they are destroyed to a large extent when treated with chromium, valuable new chromium compounds have been obtained. In the course of time a complete range of new *o*-oxyazo-dyestuffs was produced which, when converted in a suitable manner into chromium compounds, are excellently adapted for the dyeing of wool in an acid bath. To this series belong, *inter alia*, the chromium compounds of the *o*-oxyazo-dyestuffs built up by the use of sulphamides as diazo- or coupling components. New *o*-oxyazo-dyestuffs were produced from amino-naphtholdisulphonic acids, which, in the form of their chromium compounds, have proved exceptionally valuable. The same refers also to the chromium compounds of a series of new pyrazolone and acetoacetanilide dyestuffs.

Neolan Colours for Wool Dyeing

Work extending over several years produced a range of about 30 Neolan colours, comprising all the shades of the spectrum. By their introduction the old problem of wool dyeing, to produce in an acid bath, without after-treatment, thus preserving the fibre to the greatest possible extent, very pure shades of great fastness to light, milling, washing, decatising, acid, and perspiration, has been solved. In more recent times the class of Neolan dyestuffs has advantageously been supplemented by Neolan Black WA; in this colour we have the first black acid dyestuff of commerce which, besides possessing great beauty and easy applicability, exhibits substantially the properties of the black after-chroming dyestuffs. Further, metal compounds of azo-dyestuffs were recently produced in which several dyestuffs are intermolecularly bound to the same metal complex.

These Neolan colours and their application, as well as the intermediates used in their manufacture, are protected by numerous patents in all important countries, and on the same basis as the Neolan colours a series of valuable dyestuffs for dyeing viscose has in recent times been brought on the market under the name of Rigan colours.

The observation mentioned previously, that chromium compounds of azo-dyestuffs are suitable for dyeing wool in an acid bath, has also inspired the production of a series of other metal compounds of azo-dyestuffs containing groups capable of being chromed. Thus, our Society produced a range of copper compounds of *o*-oxyazo-dyestuffs dyeing from an acid bath, which have become known under the name of Lanazol colours, and which yield shades fast to light and washing on wool and other animal fibres.

Metalliferous Dyes

In further prosecution of the investigations on metal compounds of azo-dyestuffs, our Society has discovered and patented new *o*-oxyazo-dyestuffs which have an affinity for cotton and yield very fast shades, particularly to light, when subsequently treated on the fibre with metal salts, especially copper salts. These metalliferous dyes can also be produced in substance, prior to the dyeing process, according to processes patented to the Society. They represent an excellent new class of direct-dyeing dyestuffs for cotton, which belong to the most important members of the well-known direct dyestuffs fast to light of our Society, the so-called Chlorantine Fast Colours, such as, *inter alia*, Chlorantine Fast Violet BLN, 4BLN, RL, 2RL, Chlorantine Fast Brown BRL, 3GL, 8GL, Chlorantine Fast Rubine RL, and so on. The Chlorantine Fast Violet brands were the first direct violets of the market, excellently fast to light and at the same time vivid.

The range of the Chlorantine Fast Colours was advantageously extended by manufacturing further red, yellow, brown, orange, black, but especially a number of very pure blue types of excellent fastness to light, such as Chlorantine Fast Blue GL, 2GL, 4GL, GLN. In most recent times there has been found and introduced a dyestuff, Chlorantine Fast Blue 3GLL, which must be regarded as the direct blue fastest to light on the market.

Introduction of the Cyanuric Residue

An invention of particular importance was the introduction of the cyanuric residue into the molecule of azo-dyestuffs. This may, for instance, be effected by treating the finished azo-dyestuffs, or the corresponding intermediates, with cyanuric chloride. Of the extensive range of cyanuric azo-dyestuffs obtained in this manner, which are protected by numerous patents, some direct-dyeing dyestuffs have proved of particular value and have extended the range of our Chlorantine Fast Colours most advantageously; for example, Chlorantine Fast Green BL, the first pure green direct dyestuff excellently fast to light, further the greenish-blue Chlorantine Fast Blue 8GL, the Chlorantine Fast Red 5GL excellently fast to light, and so on.

It is not only in direct colours that the introduction of cyanuric residue has yielded very fruitful results. By this reaction, dyestuffs are also obtained which can be coupled on cotton with diazotised *p*-nitroaniline, giving brown, green, etc., shades which can be discharged. By a corresponding improvement of the reaction it was further possible to produce direct and acid colours, red, blue, and violet chrome printing colours, diazotisable and developing colours of all shades, exhibiting excellent fastness properties.

In the vast field of dyestuffs brief mention may also be made of the ranges of Kiton Colours, Kiton Fast Colours, Cloth Fast Colours, After-chroming Colours (among the latter particularly valuable new black brands), Chrome Printing Colours, Developing and Diazotisable Colours, Polymethine Colours, Sulphur Colours, and the Leather Finish Colours developed in the last years with success. A group of dyestuffs worked out and placed on the market lately is formed by the Cibacet Colours for dyeing acetate silk. They belong to the range of water-insoluble unsulphonated azo- and anthraquinone dyestuffs, and are on the market in the form of excellently soluble powders in a highly dispersed form.

Manufacturing Facilities

The extensive inventive activity of our firm in the most various fields of the dyestuff chemistry, briefly sketched in the foregoing, which culminated in the creation of important ranges of highly valued patented classes of dyestuff specialities (among which the Ciba and Cibanone dyestuffs and the Chlorantine Fast- and Neoian-Colours are to be considered as the most important) and the considerable development of these dyestuff classes, has involved a considerable extension of the manufacturing plant in our Swiss works, which was effected on the

latest experiences of the technology in respect to apparatus and machinery according to the laws of economy and expediency. For rational production of all the vat dyestuffs and the appertaining intermediates extensive new manufacturing plants have gradually been erected since the war. In conformity with the development of the vat dyestuff department, also the production of pure anthracene and anthraquinone, as well as also of alizarin and its derivatives, was taken up on a large scale. In connection with the production of pure anthracene that of pure carbazole was also started.

The considerable development of the azo-department involved the erection of extensive new premises for the azo-plant. For the most important intermediate products of the naphthalene and benzene series spacious new manufacturing plants were erected already a number of years ago, which, in the meantime, have been substantially enlarged, and supplemented just recently by a further modern large plant. Further, in the last years various important auxiliary plants have been considerably enlarged and modernised. Thus, for instance, quite a modern high-pressure steam-generating plant was erected; further, an oleum plant of the latest approved system, a large central storehouse, and new laboratory buildings. The existing dyehouse and the ice plant were considerably extended. The older manufacturing plants were modernised, with considerable capital expenditure.

Auxiliary Products for the Textile Industry

In the field of the auxiliary products for the textile and dyeing industry our firm has also gained a footing in recent times, and a series of patented products are already on the market or about to be issued, *i.e.*, some wetting-out agents both for vegetable and animal fibres for acid, neutral, and alkaline milling, for mercerisation and carbonisation; washing agents for wool; an agent for mordanting cotton for basic dyestuffs; an agent for reserving wool; emulsions for impregnating and waterproofing textiles; agents for preventing the precipitation of lime soap; an intensifier for printing; emulsifiers for various purposes. I should particularly like to emphasise the important discovery of the Sapamines by our firm (basic derivatives of higher fatty acids), whereby the field of the acid soaps was opened up. The Sapamines are, *inter alia*, adapted for washing animal fibres, for wetting textiles, for emulsifying, and for rendering direct dyeings fast to water.

In the department of textile chemistry introduced in recent times a series of scientifically interesting and technically promising new processes, both for the production of new immunised yarns and for the manufacture of new cellulose derivatives, have already been discovered and patented. Thus, a new process for the immunisation of the cellulose was found, for example, by causing cyanuric chloride to react on cotton in its various stages of working up. Further new and characteristic processes for acetylating cellulose xanthate and regenerated cellulose have been found and applied for patent protection, which lead to spinning fibres exhibiting increased solidity as compared with viscose, and essentially increased stability towards heat, particularly as compared with acetate silk, and in connection therewith a materially improved fastness to ironing. Finally, a new solvent for cellulose has been found which permits the dissolution of cellulose without alteration. On the basis of this new solvent it is possible to convert not only cellulose in its different forms, but also cellulose and silk wastes, silk fibroin, etc., into new spinning fibres.

Production of Dyestuffs in France

ACCORDING to preliminary figures, French production of dyes in 1930 was about 500 metric tons less than the 1929 output, accompanied by similar decreases in imports and exports. Comparative data in metric tons for recent years are shown below:—

	1928. Tons.	1929. Tons.	1930. Tons.
Production	15,603	16,431	15,950
Imports	1,559	1,518	1,362
Exports	3,653	3,066	2,217

Detailed production figures during 1930 for the production of aniline dyes totalled 6,610 tons; vat and indigo dyes, 4,250; sulphur dyes, 2,410; diphenyl and triphenyl methane dyes, 1,085; Alizarine dyes, 610; Indophenol azines, oxazines, thiazines, 115; others, 870 tons.

Formic Acid in the Dyeing Industry

By K. L. Schanbacher

This article shows how formic acid combines the advantages of acetic acid with the economy of sulphuric acid; it is the essence of a paper recently read before the American Association of Textile Chemists and Colourists.

In practice, with the average acid colours, the ratio of 1 lb. of 90 per cent. formic acid is equal to 8 lb. of 28 per cent. acetic or 4 lb. of 56 per cent. acetic. This ratio is not a fixed and firm thing and is subject to slight variations depending upon the colours and other conditions. From the above ratio it is therefore quite apparent that formic acid is more economical than acetic acid.

The next and most important consideration is the quality of work it produces. The two conditions that every dyer aims to obtain are level shades and soft, easily wound skeins. For level dyeing on most types of yarns and pieces formic acid gives excellent results with the average run of acid colours. If acetic acid is used the exhaustion is incomplete unless a very high percentage of acid is used. This generally increases the cost beyond the allowed rate. Even then, to obtain a shade which will look well-covered and penetrated, a stronger acid must be used. If sulphuric acid is used after partial exhaustion with acetic acid it may exhaust the bath too quickly and force all the dye on to the outside of the fibre. Prolonged boiling is then necessary for proper levelness and penetration. This may also cause the shade to boil duller and heavier, resulting in a poor match or a rejected lot. If the lot is started carefully with formic acid and exhaustion completed by well-diluted additions, as a general rule the batch will be finished more quickly, the shade will be brighter and fuller, and the exhaustion accomplished more economically than if a combination of acetic and sulphuric acids are used. In the case of yarns a swelling action takes place and makes the skeins fluffier and softer to the feel.

Quicker and More Uniform Penetration

For the very sensitive colours acetic acid is undoubtedly the best medium. However, there are a wide range of colours that come under the classifications of moderately sensitive and insensitive on which formic acid can be used to best advantage. The reason for this is that formic is a stronger acid and gives a more complete exhaustion of the dyebath, yet is not so strong that the colours will jump, resulting in unevenness.

Another virtue of formic acid is that the penetration will be quicker and more uniform. This is due to two factors, viz., the greater strength of the acid and because formic acid has the unique characteristic of having the properties of an aldehyde besides those of an acid. This is due to the fact that the aldehyde group is contained within the formic acid molecule. It seems that the aldehyde group within the formic acid molecule has the peculiar property of swelling animal fibre. The average moderately sensitive colour can be dyed to better advantage with formic acid because of greater levelness and better penetration, but should there be any question in the dyer's mind of how a particular colour will take, using formic acid, it is suggested that the acid is fed on gradually, obtaining the same effect at the beginning of the bath as when using a weak acetic acid solution.

It often happens that the dyer wishes to use a combination of colours calling for a very sensitive and a moderately sensitive dye. In such cases formic acid fits in beautifully. Acetic acid is used at the beginning of the bath to put on a sensitive colour and followed with gradual addition of formic acid to put on an insensitive colour. Here the advantage over sulphuric acid as the secondary acid lies in the fact that should there be any of the sensitive dye left in the bath the formic acid is not so strong that it will cause it to jump on the fabric with resulting unevenness. Also when using sulphuric acid in such a combination of colours it is very difficult to obtain proper penetration. In order to obtain a penetration of proper levelness with a mineral acid it is necessary to keep the bath at the boil a long time, which is not so very desirable, or use excessive quantities of Glauber's salt which, although cheap, is an added expense, and then on drying the material one can never be positive that it will not show the tendering effect of a strong mineral acid.

Silvertone Effects

Recently, too, there has been a great demand for a mixture

of wool, rayon and cotton where the cotton or rayon is left white and the wool is dyed. These are known as silvertone combinations. Where acetic acid is used as the exhausting agent the rayon or cotton is generally stained slightly or heavily, depending on the types of dyes and the percentage of acetic acid used. Some dyers have finished their dyeing by feeding on vitriol at the end of the operation. This can be used with little danger of harming the cotton or rayon providing one or two thorough washes are given at the end. This worry and extra work is entirely eliminated, however, when formic acid is used, for the goods can be taken from the dyebath and dried hot, if necessary, and no tendering action will take place. The formic acid is volatile and as the moisture is dried off formic acid is carried with it. In the case of sulphuric acid there is an increase of percentage of acid as the moisture is driven off, resulting in complete carbonisation of the vegetable fibres where washing is neglected.

Recalling the swelling effect of formic acid on the animal fibres, it is this same characteristic of formic acid that makes it a valuable scrooping agent, and in this connection has found favour with many dyers. It is as efficient as either tartaric or citric acids and has the advantage of being considerably lower in cost. The usual practice of scrooping is to run the dyed lot into a warm bath containing soap, then transferring the lot into a cold bath containing 1 to 2 or even 3 per cent. of formic acid, leaving this run for about 15 minutes, after which the material is removed and follows the ordinary procedure. No washing off is required.

Advantages in Chrome Dyeing

One of the most important uses for formic acid is in chrome dyeing. The advantage of using the material in this process again lies in the fact that it has a swelling effect on the fibre and is a strong reducing agent. Its reducing action combined with the swelling effect makes it possible to use less chrome in the fixation of the colour, and to accomplish this operation in a shorter length of time than with either acetic, sulphuric acid or argols. Here again a very substantial saving can be effected, both in the amounts of acid used and in the reduced amounts of chrome required, not to mention the elimination of argols from the formula. When using formic acid there is a distinct advantage over the tartar process because practical tests have shown that the use of tartar is accompanied with a harsh wool.

It might be of interest here to describe some tests made in the chroming department of a very large woollen mill. In this chroming department they had a large number of Hussongs and were using approximately the following formula: 20 pints acetic acid 56 per cent. concentration, 8 pints vitriol, 5 lb. of crude grade argols, and 10 lb. chrome. The type of the material was woollen raw stock. In order to make the comparison as accurately and fairly as possible two machines were selected, one of which they ran by their method and the other was turned over to us. Our formula called for 4 pints of 90 per cent. formic acid, 2 pints vitriol and 5 lb. chrome. In their procedure they added the total quantity of acetic after the bath had reached 100° F. We added 4 pints of well-diluted formic acid at the same temperature, the bath was brought to the boil and boiled for 20 minutes, after which time 2 pints of well-diluted vitriol was added. Boiling was continued for 15 minutes, and then 5 lb. of chrome was added. The lot was then boiled for ¼ hour longer and plugged.

From the standpoint of economies effected the important thing to observe is the substantially reduced amounts of chrome used. In this particular case one-half the amount that was used when acetic acid had been employed. Another saving effected was the elimination of argols, not to mention the reduced acid costs. Basing the figures on the price then in effect, a saving of almost 50 per cent. was effected with the same quality of workmanship, if not better. Of course, other woollen plants may use different procedures wherein the saving would not be as great as in this particular case. In most cases, however, at least a 10 to 20 per cent. saving could be effected.

Cross Dyeing and Fulling

Formic acid can also be used to excellent advantage in cross dyeing. Many fabrics are made of cotton and worsted, the cotton being dyed in the skein and then woven with the white worsted which must be dyed differently and usually in a brighter shade. In numerous instances in this type of work vat and developed colours are used. Due to the lower cost of developed colours they are preferred to the vats. It has been found that the longer the coloured cotton is boiled in the presence of wool in an acid bath the lighter the shade of the cotton will be. This in turn tints the worsted material and prevents the production of clear, bright colours.

Where coloured pieces which have been dyed with regular acid colours are fulling with soaps it often happens that the alkali present neutralises the acid contained in the yarn which is holding the dye to the fibre. This results in bleeding of the colour and in some instances a complete change of shade. Occasionally, if pieces of different shades are being fulling at the same time, the dye released by the alkali stains the other shades present and causes unexpected difficulties. If these same pieces are fulling with formic acid at the rate of 2 to 3 per cent. diluted with water, the colours will not bleed off and will remain clear and bright. A light fulling can also be accomplished quicker with formic acid than it can with soaps. This is probably due to the formic acid swelling the fibres and making them stick out and become attached to other fibres as they are forced through the fulling mill. Due to the present demand for a low-priced cloth that will feel as thick and full in body as the higher-priced fabrics, many manufacturers are making their cloth from low-grade yarns and then fulling to give the cloth the required body. Another advantage of fulling with formic acid instead of soap is that it eliminates all danger of the for-

mation of insoluble calcium or magnesium soaps due to hardness of the water.

Use of Formic Acid Prior to Weighting

There is still another use for formic acid which is being developed in this country at the present time and which is at present used in Switzerland with great success, viz., as a wash before weighting with tin salts. In this process the material is washed before tinning in a slight concentration of formic acid, the idea being that the formic swells the fibres and makes the penetration of the tin much easier and quicker. It is claimed that instead of having to give the skein three dips it can be tinned with one dip. The exact details of this process are not in my possession, and as far as I know this process is merely in the experimental stages in this country. However, there are several companies that are trying to develop it and it is no doubt merely a question of time until detailed information on this process will be available.

In the silk industry formic acid finds practically the same uses as in the woollen industry. Naturally, slight modifications have to be made, and generally it is necessary to use slightly higher concentrations of formic acid than in wool dyeing. In silk dyeing, when the dyes are exhausted with formic acid they go on more slowly than when either sulphuric or acetic acids are used. It has been found that the exhaustion is better, and consequently the usual after-washes are reduced to a minimum. This in itself is a decided economy, not to mention the usual saving in acid cost effected by the use of formic acid. The outstanding points to remember when using formic acid are that it is an acid stronger than acetic and about on a par with sulphuric acid, and that it has swelling and also reducing properties.

Dyes and Their Application: Recent Technical Progress

By L. J. Hooley

Insoluble Azo Colours

This branch of the azo colours still retains its interest as being the one in which the most important developments are taking place. Very recently the first of the greens of this series, Duratol Green G, was placed on the market by Imperial Chemical Industries, Ltd.

In previous issues of *THE CHEMICAL AGE Monthly Dyestuffs Supplement*, these insoluble azo colours have been discussed largely from the point of view of synthesis. In the following some recent developments and publications dealing with application and other aspects will be reviewed.

Application

The normal method of application is not generally suitable for dyeing wool both because of the alkali problem and because the sequence of operations in dyeing and finishing depart somewhat from those generally applied to wool. As far as this latter particular is concerned, silk is better adapted. Devices for using naphthols under mild alkali conditions include the use of an alkali-starch compound with a phenolic coupling compound. As an example the o-toluidide of 2 : 3-oxy-naphthoic acid is incorporated with 3-8 per cent. of alkali starch, and the resulting preparation used in the padding bath (B.P. 325,563, I.G.F.A.-G.). This idea of using alkali-starch may be regarded as an extension of the same process with vat colours. Apart from the advantage as regards the low alkalinity of the bath required, the products have greater stability.

Mono- and di-aryldes of 2 : 3-oxy-naphthoic acid in which the arylide group is not substituted by hydroxyl have capacity for being absorbed by wool from a bath of low caustic alkalinity (B.P. 310,758 S.C.I.B.). An alkali content of 0.6-1.8 gm. of caustic soda per litre is recommended, this giving bright shades.

J. A. Wallwork (*J. Soc. Dyers and Col.*, 1930, 10) details suitable methods and machinery for wool either alone or in unions and also gives rules for controlling the depth of shade in the case of the latter. H. Lederer also discusses the application of the Naphthols to wool in the *Textilberichte* (1931, 112). Also in *The Dyer* (1931, 607) H. Blackshaw describes the dyeing of the Duratols.

Aromatic amino-sulphonylamides are suitable for animal

fibres as they can be washed after padding. As an example o-amino-p-toluenesulphonamide may be used along with 1 : 5-dihydroxy-naphthalene (B.P. 315,899. Ver. f. chem. u. met. Prod.).

The Naphthols may be used in conjunction with aniline black by printing the fabric with, for instance, the anilide of 2 : 3-hydroxy-naphthoic acid after padding with the aniline, aniline hydrochloride and oxidising agent. Along with the anilide, zinc oxide is used together with sufficient alkali to neutralise the aniline of the aniline padding. After steaming the cloth is passed through the requisite diazo solution (B.P. 305,476, I.G.F.A.-G.). For use with vat colours the fabric is padded with an arylide, dried and printed with formaldehyde hydrosulphite and a vat dye and after a second drying, steaming and also passing through a diazo solution. A method of avoiding loose colour on the fibre when machine dyeing material in compact form is described by F. Karrer in B.P. 338,012, this involving a drying in the machine between the padding and developing.

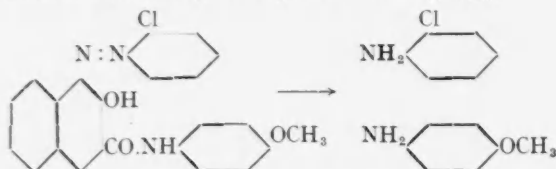
Preparations of the Rapid Fast series containing both components can be made by grinding a nitrosoamine salt with an arylamide free from carboxyl and sulphonic groups and a substance which will absorb water: dry powders can be obtained (B.P. 340,534, I.G.F.A.-G.).

Kier Boiling

F. M. Rowe has published some interesting results on the action of caustic soda on insoluble azo colour on the fibre which throw light on the kier boiling process. As an example a 2.9 per cent. dyeing of Fast Yellow G Base coupled with Naphthol AS-RL after boiling for 6 hours with a 0.36 per cent. caustic soda solution appeared almost stripped of colour. In spite of this 1.7 per cent. still remained on the fibre but was aggregated into different physical form. Further 0.78 per cent. of unaltered colour could be separated from the fibre by squeezing, showing that this had merely been loosened. The remaining colour was found to be destroyed partly by reduction of the azo group and partly by hydrolysis of the arylamido group, chloroaniline and p-anisidine being recovered from the liquor.

In couplings with nitro groups present these were found to

be partly reduced to amino groups. The reducing agent is cellulose in the presence of caustic soda together with impurities in the cotton (*J. Soc. Dyers and Col.*, 1931, 35).

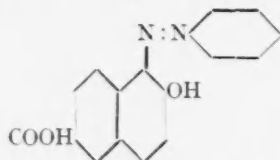


Another paper by the author in the same issue describes the tests carried out for the I.C.I.-I.G.F.A.-G. Naphthol Revocation case.

Coupling with Alkyloxy Naphthoic Acids

In the coupling of ethers of naphtholcarboxylic acids, as distinct from the hydroxy bodies, G. B. Jambuserwala and F. A. Mason have coupled 2-methoxy-3-naphthoic acid with diazotised p-chloroaniline, dichloroaniline, p-nitroaniline and some other bases; although earlier work by Meyer (*Annalen* 1920, 97) suggested that coupling did not take place with ethers. In some cases complete or partial loss of the alkyl group occurred. (*J. Soc. Dyers and Col.*, 1930, 339.)

While dealing with naphtholcarboxylic acids as distinct from their arylides, mention may be made of (U.S.P. 1,709,989, Grasselli) where 2:6-dihydroxynaphthoic acid is coupled with diazo compounds. Aniline gives a yellow-red and 5-nitro-o-aminophenol a brown. The dyeings may be chromed.



An interesting development of the application of insoluble azo colours is that of B.P. 333,506-7 (S.D.L., J. Thomas and D. A. W. Fairweather) where the coupled dyestuff is employed in soluble form by using as one component a sulphuric ester of a reduced quinone such as anthraquinone. The ester group confers solubility and is removed after impregnation by acid oxidation.

As regards naphthols other than the well-known ones of the 2:3-hydroxynaphthoic arylide type, the following recent developments, dealing with related ones, may be mentioned. In B.P. 326,971 (I.G.F.A.-G.) naphthols are made by treating 2:6-dihydroxy-3-naphthoic acid with ammonia or a primary or secondary amine; substitution takes place in the 6 position. When the substituent is an arylamino body, boiling with bisulphite solution and a p-aminophenol, causes replacement of the 2-hydroxyl group by a p-hydroxyarylamino group while the carboxyl group is lost (B.P. 334,700, I.G.F.A.-G.).

Scottish Dyestuffs Market

THERE is not much new to report as regards business during July, which has remained much the same as in the month immediately preceding. The holidays have, of course, caused some interference, but it has generally been found possible to make up for lost time or partly to make it up by working on full time during the remainder of the month.

On several occasions attention has been drawn to the enterprise shown by the woollen manufacturers of Scotland; although during the last two years or so conditions have not been anything like so prosperous as they were immediately prior to this, there has been a very determined effort made all along to maintain the level of business as far as possible. Fairly recently deputations have been sent to the United States and to Europe to get into more intimate touch with the important buyers there. It is now understood that the Scottish woollen manufacturers have decided to join together at next year's British Industries Fair and to have an impressive exhibit which will adequately display the high quality of Scottish tweeds.

In the Glasgow district, dyers have not been very fully occupied. Dyestuffs sales and chemicals show no change.

Dyes and Textiles

Notes on Current Researches

CELLULOSE BUTYRATE.—Although cellulose acetate is almost the only cellulose ester rayon at present being produced in commercial quantity, there is reason to suspect that in the future a considerable amount of rayon may be manufactured from cellulose butyrate. At the moment, therefore, this little known ester of cellulose has speculative possibilities and it is useful to note a paper by G. v. Frank and H. Cohn (*Cellulosechemi*, 1931, 12, 68), which describes a comparatively simple method of preparing cellulose butyrate and summarises many of its properties.

Apparently it is convenient to convert the raw cellulose material into the formic acid ester and then further convert this to the butyrate rather than attempt the direct formation of the cellulose butyrate. Thus the mode of preparation may consist of steeping cotton in 80-90% formic acid at 20° C., for one day, then press out excess of acid and remove the remainder by thorough washing with water; afterwards the product (cellulose formate) is dried in air and then treated with a mixture consisting of zinc chloride, butyric acid anhydride, and butyric acid at 40° C., whereby a cellulose ester of both formic and butyric acids is formed. This mixed ester can then be heated at 90° C. in 93% butyric acid whereby the formic acid residues are displaced and cellulose butyrate itself obtained.

Rayon made from cellulose butyrate resists saponification by alkalis much more than does cellulose acetate rayon; it is also much more ductile after having been stretched until the elastic limits have been exceeded.

BLEACHING WITH ORGANIC PER-ACIDS AND PEROXIDES.—Although the use of solutions containing active chlorine (bleaching powder or sodium hypochlorite) for the bleaching of rayon materials is almost universal, it would appear from U.S.P. 1,767,543 that certain advantages are obtained when bleaching agents containing active oxygen are employed. It is known that in a rayon material the cellulose is very reactive and can therefore be easily degraded during bleaching with consequent loss of strength and elasticity. Such deleterious effect on the rayon can, it is claimed, be avoided if cold or slightly warm solutions of organic per-acids or peroxides under neutral or slightly acid conditions (in contrast to the slightly alkaline conditions usually employed with hydrogen peroxide bleaching liquors) are used.

Among the bleaching agents thus found suitable are benzoyl, phthalyl, acetyl, succinyl, maleic, and fumaric peroxides, and also the corresponding per-acids. About 1.5 grams of benzoyl peroxide in 5 litres of water is found sufficient to bleach 1 kg. of rayon, and similar quantities of the other bleaching substances are suitable.

MANUFACTURE OF INDANTHRENE BLUE.—Indanthrene Blue formed by the alkali fusion of beta-aminoanthraquinone is a useful dye itself and also because on chlorination it yields blue vat dyes having excellent fastness to bleaching. It is now found by T. Maki (*J. Soc. Chem. Ind., Japan*, 1930, 33, 456B) that the presence of phenol in the caustic potash melt accelerates the rate of formation of the dye although it does not increase the yield. Maki indicates that the highest yield of Indanthrene Blue is obtained by carrying out the fusion in the presence of phenol at 210° C. for 1 hour; if a higher temperature is employed there is danger of the blue dye formed being further decomposed by the caustic potash present.

DELUSTERING CELLULOSE ACETATE RAYON WITH ZINC SULPHIDE.—The production of low lustre rayon is very important since a large proportion of the rayon garments now being manufactured must conform to the fashion which abhors bright lustre. It is evident from the patent literature that textile technologists are fully alive to the demand for low lustre rayon goods, for quite a large number of delustering processes have been recently protected.

Egyptian Dye Industry to be Modernised

A REPORT outlining steps for the improvement of the Egyptian dye industry was recently approved by the Consultative Committee of the Egyptian Department of Commerce and Industry. The report includes plans for the construction of a modern dye factory.

Dyestuffs Monthly Supplement

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Better Dyestuff Exports

It is encouraging to note that while so many branches of trade have experienced a decline in exports, the exports of British dyes and dyestuffs, principally those derived from coal tar, have undergone an improvement in the first seven months of this year. Comparing July, 1931, with July, 1930, we find that the quantities of dyestuffs exported have increased from 14,385 cwt. to 19,619 cwt., not much below the figure of 1929, while the value of the dyestuff exports has increased to £101,549, from £93,672 in 1930, and £95,345 in 1929.

This improvement in the month's figures is fully confirmed by the returns for the seven months ended July 31. The actual quantities exported have declined from 122,413 cwt. to 115,383 cwt., although in coal tar dyestuffs there was an increase from 63,002 to 71,461 cwt. over the figures for the corresponding period of 1930. In values, however, the dyestuff exports have advanced to £658,579 for the seven months of this year, as compared with £577,765 for the first seven months of last year, and £645,359 for the corresponding months of 1929. These figures indicate that the industry, judged by money values alone, has grown into a substantial national asset, although its importance remains even greater as an essential contributor to the textile industry and as a practical school of organic chemistry. The record for this year up to date is distinctly encouraging.

A Dyestuff Pioneer

Mr. Jacob F. Schoellkopf, the donor of the Schoellkopf gold medal just awarded to Mr. Frank J. Tone, for valuable work in metallurgy, by the Western New York Section of the American Chemical Society, has long been identified with the production of aniline dyes in the United States. Entering the aniline dye business in Buffalo in 1880, he foresaw the possibilities of dye manufacture in America. His company, incorporated in 1900 as the Schoellkopf Aniline and Chemical Works, with himself as president, became the first in the United States to specialise in the manufacture of dyes for industrial purposes. Nationwide demands for American dyes, created by the World War when the United States stopped importing dyes from Germany, led to a more extensive manufacturing programme, and the Schoellkopf Aniline and Chemical Works, to expand their facilities, merged with the National Aniline and Chemical Co., which name it now bears. While developing the aniline colour industry, of which he is regarded by chemists as the founder in the United States, Mr. Schoellkopf was also interested in the development of electric power. As early as 1900 he established a connection with the Hydraulic Power Co. of Niagara Falls, which was absorbed in 1917 by the Niagara Falls Power Co. Resigning as president of the National Aniline and Chemical Co. in 1918, Mr. Schoellkopf became chairman of the board of the newly formed Niagara Falls Power Co.

Two New Blacks

Included in a batch of new colours just placed on the market by Imperial Chemical Industries are two notable blacks—Coomassie Fast Black GS, an addition to the firm's acid colours and described as "an I.C.I. speciality possessing several interesting features," and Altex Union

Black DBN, an important addition to their range of Altex Union colours.

Coomassie Fast Black GS gives greenish-black shades of very good fastness to washing, milling, perspiration and light. It is extremely suitable for dyeing hosiery yarns, yarns for bathing costumes, slubbing and loose wool, whilst its good solubility and good level dyeing properties make it of particular interest for piece dyeing. Although the colour is closely related in general properties to the well-known Coomassie Fast Black BS type, it is considerably more level dyeing and gives excellent results even when dyed from a sulphuric acid dyebath. This colour is of interest for dyeing wool felts, and is a valuable alternative to the ordinary acid black brands for those cases where a higher degree of fastness to light and water is desired. It possesses excellent affinity for the wool fibre when dyed from a neutral dyebath, and therefore is specially interesting for the dyeing of union materials. It is eminently suitable for application to pure silk either weighted or unweighted, for which purpose it demands special consideration. It is suitable for the printing of wool, carpet yarns and pure silk.

Altex Union Black DBN, in common with the other members of the Altex Union range, dyes mixtures of all the various textile fibres—cotton, wool, silk, viscose and acetate silk—in solid shades from a neutral dyebath. This new brand represents a great improvement on the older and well-established colour, Altex Union Black DB, in that it produces solid blacks on garments varying widely in the proportion of the several fibres present. Altex Union Black DBN is first stirred to a smooth paste with cold water and then dissolved by the addition of boiling water. The solution obtained should be added to the dyebath through a fine sieve.

A Brilliant Acid Scarlet

A very interesting further addition to the I.C.I. acid colours is Coomassie Red PGS, the brilliant tones of which are well exhibited on botany slubbing, hosiery yarn, tin-weighted silk, and gloria. This new colour is particularly suitable for the production of bright scarlet shades, of very good fastness to washing, light and sea water, on yarns and slubbing for use in the bathing costume trade, and also is of considerable interest to the hosiery and fancy worsted trades. It is particularly suitable for the dyeing of wool generally on account of its very good fastness to milling and carbonising. This dyestuff possesses good solubility and may be applied in the various types of circulating machines; it is specially suitable for dyeing both weighted and unweighted silk, producing brilliant scarlet shades of good fastness to water, washing and light. It may be applied from a neutral soap bath, and its affinity for tin-weighted silk makes it of particular interest for the dyeing of crêpe materials. The colour possesses very good affinity when dyed from a neutral dyebath, and is of interest, therefore, for the dyeing of wool and silk in union materials. Solid shades are obtained on wool and silk mixtures whether dyed from a neutral or mildly acid dyebath. It is suitable for the direct printing of wool and silk, and is dischargeable with hydrosulphite, and also for the production of bright scarlet shades on both vegetable and chrome tanned leathers. Coomassie Red PGS possesses excellent

affinity for the wool fibre when dyed from an acetic acid dyebath.

Three Naphthalene Leather Browns

The I.C.I. range of acid colours for the leather trade is now enlarged by the addition of three new brands of Naphthalene Leather Brown—namely, OD, OT and OYT—all of which are said to possess excellent solubility and to be suitable for brush-stained work and for the dyeing of vegetable, semi- and full-chrome leathers. Naphthalene Leather Brown OT is particularly suitable for brush-stained work on upholstery leathers, giving a full tan shade when two coats of a solution containing 1 lb. per 10 gallons of water are applied. When applied by the usual dyeing method the shade is a neutral brown of interest to the clothing and shoe trades. The OYT brand yields shades somewhat greener in tone than OT, and provides a ground shade of special interest for antique effect upholstery leather and other brush-stained work. The OD brand, by reason of its shade, may not be of equal importance to the above brands for brush-stained work, but it is eminently suitable for the dyeing of full chocolate browns on clothing, shoe and fancy leathers. The patterns shown have been produced by brush staining on furniture hide and the paddle dyeing of sumac tanned skivers. For the stainings, two coats of solutions containing 1 lb. of dyestuff per 10 gallons of water were applied to the leather. In the case of the dyeings on skiver, dyeing was conducted in the paddle using 5 per cent. of dyestuff (calculated on the dry weight of the leather). After working for 15–20 minutes 3 per cent. of formic acid was added to aid exhaustion. These quantities correspond to 6½ oz. of dyestuff and 4 oz. of formic acid per dozen skins, averaging 10½ square feet.

A Chlorazol Bordeaux

Chlorazol Bordeaux 6BS, an addition to the I.C.I. range of Chlorazol dyestuffs, is described as suitable for dyeing cotton pieces, warps and yarns, being extremely level dyeing and noteworthy on account of its clarity of tone when dyed in deep shades. Its most important and valuable application is in union dyeing, as it is eminently suitable for dyeing the cotton in union materials. Its very good affinity at low temperatures, clarity of tone, and excellent levelling power makes it of special interest for garment dyeing and union dyeing in general. It is very suitable for dyeing jute and allied fibres, as it gives good colour value combined with brilliancy of shade. It is also of value for dyeing straw, tagal and wood chip. It is of interest for paper dyeing, whilst its good discharge properties make it of value for calico printing as a ground shade for the production of white or coloured discharge effects.

U.S.A. Textile Colour Cards

The widened activities of the United States Textile Colour Card Association during the year 1930 were outlined by Mrs. Rorke, secretary and managing director, in her report at the sixteenth annual meeting of the Association. During the year, the Association had gained 242 new members, the largest increase since 1927, and the third largest since the founding of the Association in 1915. All the textile industries were represented in this group, as well as department stores and other retailers. Interest in the Association was constantly increasing abroad. Many of the foreign members were obtained through the interest and assistance of Commercial Attachés, and through the co-operation of the Department of Commerce and State Department at Washington, who, twice a year, send every card issued by the Association to the American consuls and commercial representatives throughout the world. During the past year, the Association manufactured 54,535 colour cards, including standard silk,

standard hosiery, as well as seasonal floss, ribbon, woollen, hosiery, shoe and leather, mid-season silk cards, garment cards and the U.S. Army colour cards. Mrs. Rorke called attention to the very active co-operation that had steadily grown between the Association and the Colour Co-ordination Committee of the National Retail Dry Goods Association, in the promotion of basic shades for store-wide co-ordination. Likewise, the movement to promote colour correlation in the department stores, through concerted industrial effort, had attracted most favourable comment from leading retailers and manufacturers.

Lecithin in the Rayon Industry

Lecithin, which is an important constituent of the yolk of eggs, was, until recently, prepared exclusively from this source. The prospect of a wider use for it in the textile industry has now come almost simultaneously with the discovery that the soya bean contains lecithin in sufficient quantity to permit its extraction at relatively low cost. This extraction process is being carried on by the Hansa Mühle G.m.b.H., of Hamburg, and for use in the textile industry the product is placed on the market in three forms. Emulex is a thick, dark brown preparation, mostly consumed in dyeing and printing operations. Lecivon is of a golden yellow colour and can be employed for all purposes. Splendicithin is in the form of a thin liquid, light in colour, especially recommended for white goods. The use of these lecithin preparations is not confined to rayons, but the products are stated to be of especial interest for the finishing of rayon yarns. Experiments show that the lustre of the material is improved and a soft handle is obtained either on yard or fabric if lecithin is added to the dyebath or in a subsequent rinsing bath. Its levelling effect on the dyestuffs is also said to be very marked. An instructive paper on the practical application of lecithin in the rayon industry was published by Professor Otto Mecheels during the early part of this year (*Melliands Textilberichte*, Feb. 1931.)

An Improvement in Re-lustering Rayon

For the purpose of obtaining decorative lustre effects on cellulose acetate rayon materials, it has often been found desirable to restore the lustre as well as to reduce the lustre of this type of rayon. Material which has been de-lusted by treatment with a boiling soap solution is often re-lusted by subsequent treatment with suitable organic solvents followed by drying. Steaming for a period of one to two hours, as described in a recent patent (E.P. 332,231), was a simpler and less costly operation. In a more recent patent (E.P. 349,900), however, it has been suggested to effect this steaming under pressure at 150° C., instead of 100° C., a procedure which is claimed to give a noticeable improvement in the final appearance of the treated material. This treatment may be accompanied by stretching, as this has a recognised beneficial influence in restoring the lustre of all types of rayon.

Dyestuff Licences for August

The following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during August has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee:—

The total number of applications received during the month was 428, of which 371 were from merchants or importers. To these should be added 2 cases outstanding on July 31, making a total for the month of 430. These were dealt with as follows:—Granted, 404 (of which 401 were dealt with within 7 days of receipt); referred to British makers of similar products, 22 (of which 20 were dealt with within 7 days of receipt); outstanding on August 31, 4. Of the total of 430 applications received, 421 or 98 per cent. were dealt with within 7 days of receipt.

Basic Intermediates for Dyestuffs: No. XLV.—Quinoline and Related Compounds—(continued)

By "Consultant"

THE previous article of this series dealt with the dyestuffs intermediates used on a comparatively large scale industrially; the dyes and intermediates of the photosensitising dyes series are only produced in comparatively small amounts, but their value is considerable since they have contributed much to the art of photography. It is convenient to divide the quinoline group of dyes into four groups according to the chemical skeletons from which they are derived. These four groups are:—

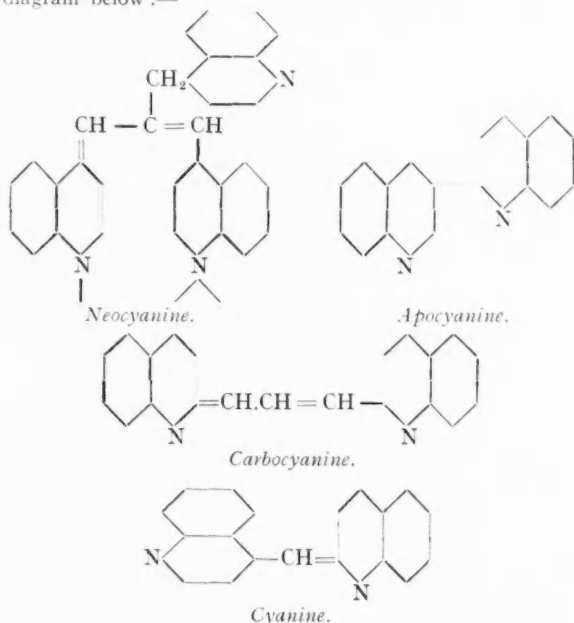
(a) *The Neocyanines*.—These are, in reality, triplepidyl methanes, whose investigation is due to Hamer.

(b) *The Apocyanines*.—Derived from the diquinolyl nuclei, and of academic interest only.

(c) *The Carbocyanines*.—This group is derived from a nucleus formed by joining two quinoline nuclei by the $=CH.CH=CH$ group, according as the latter link is in the 2:2¹, 2:4¹, or 4:4¹ positions, members of this group are known as Carbocyanines, Dicyanines or Kryptocyanines.

(d) *The Cyanines*.—These are derived from the nucleus obtained by joining two quinoline nuclei by means of a $-CH$ group. Here again, according to the position of the bridge link in the 2:2¹, 2:4¹ or 4:4¹-positions the compounds are known as Pseudocyanines, Isocyanines or Cyanines.

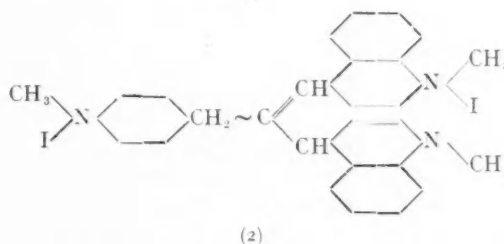
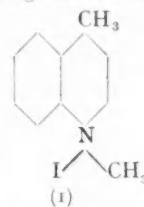
The nuclei associated with these compounds are shown in the diagram below:—



For the purposes of this article, the apocyanines and pseudocyanines may be immediately dismissed as of no commercial application. The Neocyanines are of peculiar interest in that they sensitise right up to 8000 Å, that is, further into the red section of the spectrum than any previously known dye; moreover, when a plate which has been sensitised by Neocyanine has been exposed to ammonia the range of sensitivity is increased, until it is possible to photograph light of 10000 Å without difficulty.

The preparation of Neocyanine is not difficult; lepidine is converted to its methiodide by treatment with a little more than the theoretical amount of methyl iodide. The recrystallised lepidine methiodide (1) is dissolved in hot pyridine (free from water) and the solution boiled with ethyl orthoformate when condensation takes place with the formation of Neocyanine methiodide (2). This compound is, as a matter of fact, entirely analogous to the Kryptocyanine type, save that in the latter (3) only two quinoline nuclei are involved. Kryptocyanine, now

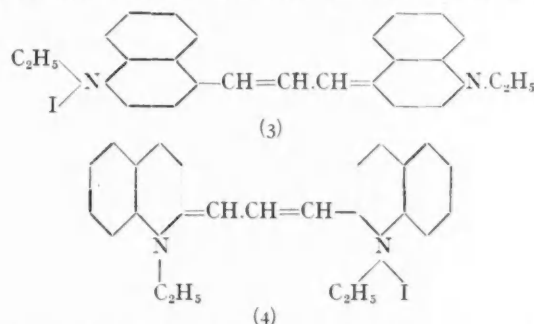
manufactured as a photosensitising dyestuff, is made by converting lepidine into its crystalline ethiodide by the action of ethyl iodide, and treating this substance with a mixture of



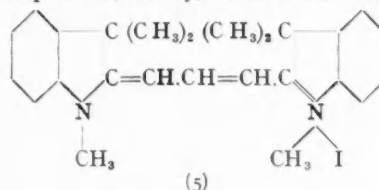
sodium ethoxide and formaldehyde. There is little doubt that it has the formula (3). A unique feature of this dye is that although it sensitises from 7000 to 8000 Å, it does not induce any sensitiveness to the green section of the spectrum; it may, of course, be used in admixture with a green sensitising dye.

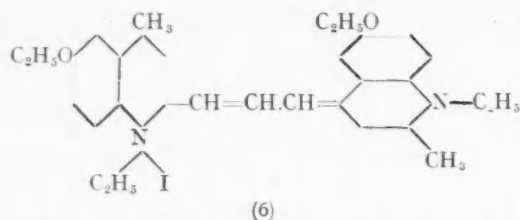
Carbocyanines

The carbocyanine group in which two quinoline residues are united in the 2:2¹ condition by the three-carbon chain is represented by the dye originally known as "Pinacyanol." Much controversy has ranged round the subject of the constitution of this dyestuff, but there is little doubt that it has the formula (4), more especially in view of the fact that it is now



made in bulk by the action of a condensing agent such as acetic anhydride upon a mixture of quinaldine methiodide and ethyl orthoformate. An even more striking improvement in the yield may be obtained by using anhydrous pyridine as a solvent. It is of interest to note that the indocyanine analogue of Pinacyanol is of value as a dye, giving a pink of great brilliance, and being known commercially as "Astraphloxine FF." Its formula is shown in (5) but it has no photosensitising properties. Of the remaining group of the cyanines one member only is of importance, namely, the so-called "Dicyanine A."



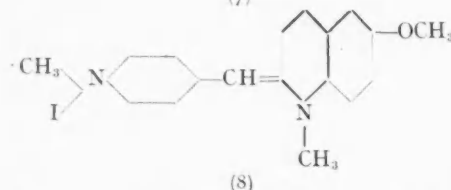
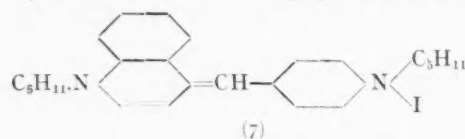


This substance can be obtained by boiling the ethiodide of 2:4-dimethyl-6-ethoxyquinoline with alcoholic sodium sulphide in the presence of a little chloroform. Its constitution, shown in (6), shows that it is a member of the 2:4-carbocyanine group. It is not a photosensitising dye, but sensitises the normal plate to infra-red rays, and is for this purpose especially valuable.

The True Cyanines

The true cyanines contain the $-\text{CH}=\text{}$ group as the link between the two quinolinium residues, and of the whole group Cyanine itself, or Quinoline Blue as it was at one time called, was the first to be discovered. It was first prepared as early as 1883 by Hoogewerf and van Dorp, by the action of alcoholic potash upon a mixture of equimolecular proportions of the isoamyl iodides of lepidine and quinoline. Its constitution is given in (7). In actual practice the derivatives of the iso-cyanine group (2:4-cyanines) have proved more valuable as photosensitising colouring matters. They are more sure in

action than the normal cyanines and are without the tendency characteristic of the cyanines of causing the effect known as "veiling." The best method for the preparation of the members



of this series is the condensation of a substituted quinaldinium methiodide with a quinolinium methiodide which may or may not be substituted. The presence of alkali is necessary to effect condensation, and the type of compound obtained is shown in formula (8).

The technical details of these condensations do not require any very specific description. They do not involve more than the simplest of apparatus and conditions. Finally, it must be mentioned that the dyestuffs of certain of these classes exert an antiseptic influence, and are therefore valued medicinally.

Dyes and Textiles

Notes on Current Progress and Research

BETA-NAPHTHOL SULPHONIC ACIDS.—A number of important acids which find a use in the manufacture of azo dyes are obtained by the sulphonation of beta-naphthol. Among these acids are to be found Crocein, G-, R-, and Schaeffer acids. The acid formed by sulphonation is obviously largely dependent on the conditions employed, but it is difficult to avoid the formation of a mixture of acids. For this reason some recent investigations by J. S. Smith, S. D. Forrester, and D. Bain (*J. Soc. Chem. Ind.*, 1930, **49**, 4101 and 4231), concerning methods for analysing such a mixture, are extremely valuable, and it is therefore interesting to note a further publication on this subject by the same workers (*Ibid.*, 1931, **50**, 1007). In addition to the above-named acids the product of sulphonation may contain oxy-Tobias acid (beta-naphthol-1-sulphonic acid), F-acid (beta-naphthol-7-sulphonic acid), and beta-naphthol-3:6:8-sulphonic acid. It is now found that bromination methods can be used for the determination of these acids, such methods being based on the observation that beta-naphthol-sulphonic acids containing a sulphonic acid group in the 8-position resist bromination in 10 N. sulphuric acid solution. The explanation of this peculiar resistance to bromination is found in the fact that in such strongly acid solution the beta-hydroxyl is sulphated and this group along with the sulphonic acid group in the 8-position exerts steric hindrance to the entry of a bromine atom in the 1-position. Full details of the conditions to be employed in the analysis are given, and it is incidentally noted that F-acid can exist as tetra-, di-, and mono-hydrates as well as in the anhydrous form.

CARBOHYDRATE ETHERS FOR SIZES.—A new departure in the preparation of sizing compositions for use with cellulose acetate rayon yarns is described in Brit. Pat. 341,516. For cotton yarns it has long been the practice to use size pastes containing starch and tallow or other softening agent, but experience has shown that these are not suitable for rayon. It is now proposed to use aqueous solutions of ethers prepared by treating starch or cellulose with alkylene oxides in the presence of caustic soda or other alkali. For example, a suitable sizing substance can be obtained by swelling starch with caustic soda and then adding propylene oxide; a reaction thereby ensues and after sufficient time the product is found to be soluble in both water and alcohol.

MANUFACTURE OF MINE-VENTILATING MATERIAL.—It is obvious that the fabric used in the manufacture of hose which is employed in the mining industry must be very

durable, and that in particular it must be waterproof and resistant to mildew and acids as water in mines is frequently acidic, whilst it should preferably be fireproof. A method for treating fabric so that it has these various qualities has been protected by U.S. Pat. 1,779,258. In the method described, cotton or similar fabric is impregnated with a composition containing crude rubber 25 parts, litharge 8 parts, sulphur $\frac{3}{4}$ part, barytes $52\frac{1}{2}$ parts, zinc salicylate 2 parts, pine tar 4 parts, paraffin $7\frac{1}{2}$ parts, and gasoline 100 parts, and then hung in a chamber for $2\frac{1}{2}$ hours at 250°F ., whereby vulcanisation of the rubber occurs. In this formula it may be noted that the litharge and sulphur are mainly concerned with the process of vulcanisation; zinc salicylate and pine tar act as antiseptics, the paraffin serves to resist acids, and the barytes confers resistance to fire.

PREPARATION OF BALLOON FABRIC.—Fabric covered with gold beater's skin is the most satisfactory material for constructing balloons and airships, because the skin covering allows only a small rate of diffusion of the gas enclosed. On the other hand, gold beater's skin is expensive, and since it can only be obtained in pieces hardly more than six or eight inches square the costs of construction when using this material are very high. Rubberised fabrics are much cheaper, but suffer from the disadvantage that they allow greater gas diffusion. Means for overcoming this difficulty, however, have now been patented (U.S. Pat. 1,779,388). The cotton fabric is first coated with a layer of rubber latex applied from an aqueous solution followed by drying, and is then successively covered with further coats of latex in which the proportion of latex is gradually diminished in favour of gelatin and glycerine as substitutes. Finally viscose film (Cellophane) is applied, and since this has good gas-diffusion resistant properties the whole fabric thus becomes highly suitable for airship construction, its manufacture being much cheaper than when gold beater's skin is employed.

German Potash Sales

SALES of the German Potash Syndicate during the past month were considerably lower than in the corresponding month of previous years, amounting to only 64,205 tons, while in August of 1930 they totalled 98,720 tons, and in August of 1929 and 1928 they had been 95,370 tons and 108,690 tons respectively. During the first four months of the current fertiliser year the sales totalled 233,231 tons.

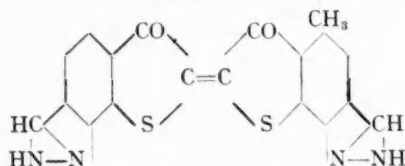
Dyes and Their Application: Recent Technical Progress

By L. J. Hooley

OUTSTANDING advances are still being made in the production of indigo dyes, as research is continually finding new products of commercial value. The fact that the whole of the indigo class of dyes are built up on the same basic type structure, makes it easier to see the difference between new products and the old products, and for the purpose of this article it will be convenient to arrange the new products according to shade.

Grey Indigo Dyes

The pyrazole ring is introduced into the indigo molecule in B.P. 345,349 (I.G.F.A.-G.). Among the examples is one which probably has the formula

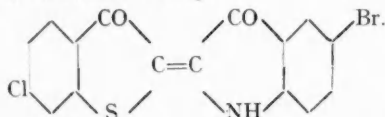


The necessary intermediate, hydroxypyrazole thionaphthene can be made by treating a 6-amino-indazole* of the following constitution:—



with chlorine and a sulphocyanide so that one X is substituted by a thiocyanato group and then proceeding *via* the mercaptan and thioglycolic acid. Most of the products are greys, probably because of their heterogeneous structure. The products are stated to have excellent fastness to light and chlorine, to be remarkably well levelling, to have equally good affinity for animal and vegetable fibres and to fix easily in printing.

Another patent for the production of a grey is B.P. 341,224 (*idem*), in this complexity is obtained by lack of symmetry both as regards the two main halves of the molecule and the substituents, the structure being:—



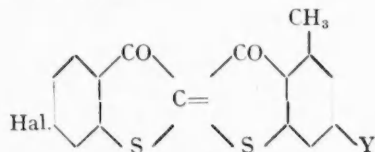
The product is stated to surpass the grey commercial vat dyestuffs hitherto known. The corresponding product with chlorine instead of bromine is, surprisingly enough, violet in colour: such a difference has not been observed hitherto.

Green-Blue Indigo Dyes

A fast greenish-blue can be obtained by condensing a reactive α -derivative of a 4-methyl-5-halogen-7-alkoxyisatin with a 4-halogen-1-naphthol (B.P. 318,107 *idem*). Instead of a 4-halogen-1-naphthol an α -hydroxyanthracene can be used, giving products analogous to Alizarine Indigo G. As an instance 4-methyl-5-chloro-7-methoxyisatin- α -chloride is heated with α -hydroxyanthracene in chlorobenzene to give a greenish-blue which is very suitable for printing. (B.P. 335,043 *idem*.)

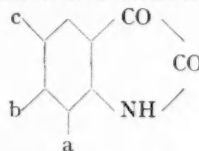
Red and Orange Indigo Dyes

Dyes of the constitution



where Y is hydrogen or halogen give pink to red shades, which while of nearly the same brightness as the known 4 : 4'-alkyl-6 : 6'-dihalogeno thioindigo are faster to light and exposure (B.P. 318,595 *idem*.)

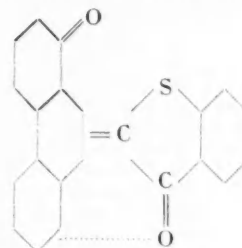
The indigo series is weak in dyes giving shades below red in depth. It is true that appropriate substituents properly placed in thioindigo have a hypsochromic effect as in 5 : 5'-dibromo-6 : 6'-diamino-2 : 2'-bis-thionaphthene indigo (Helindone Orange) and 6 : 6'-diethoxy-2 : 2'-bis-thionaphthene indigo (Helindone Orange R). Yellows have also been obtained by condensing benzoyl chloride with indigo in the presence of nitrobenzene and copper, but there is evidence that in the process the indigo structure is destroyed. Further, the indigo from 6-aminothioindoxyl and acenaphthenequinone is orange (Ciba Orange G). Indigos giving clear orange shades are now obtainable by condensing 6-alkoxy-3-hydroxy-1-thionaphthene with substituted isatins of the formula



where a is halogen or alkyl and b and c are halogen, alkyl or hydrogen (B.P. 28,741/30 *idem*).

Phenanthraquinone-Indigo

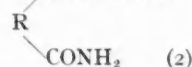
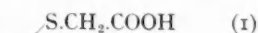
The 2-thionaphthene-9-phenanthreneindigo originally synthesised by R. Friedländer, Herzog and Voss has now been obtained in pure form by Pummerer and Luther by a variation of the original process from phenanthraquinone and 3-hydroxy-thionaphthene. The first mentioned authors recrystallised their product from nitrobenzene, but in the present paper it



is shown that this converts it to a different compound by migration of a hydrogen atom along the dotted line with formation of a bond along this line (Berichte, 1931, 831).

Reduction Products of Thioindigoid Dyes

The ordinary reduction products of indigo derivatives are the leuco bodies in which the oxygen atoms in the two ketonic groups have been reduced to hydroxyl groups, which confer solubility in alkali. New reduction products have now been obtained from certain indigos such as 4 : 4'-dimethyl-6 : 6'-dihalogenothioindigo by carrying out the reduction with a reducing agent (excluding potassium hydrosulphite) in the presence of a smaller quantity of alkali than that generally used in vatting. These reduction products are distinguished by being either entirely insoluble or only sparingly soluble in aqueous alkalis: they also differ from the leuco compounds in being stable in the presence of air or oxygen (B.P. 334,878, I.G.F.A.-G.). A later method of preparing the same bodies is that of hydrogenation with alkali and a hydrogenating catalyst (B.P. 338,104 *idem*). The products may be used for printing, the paste being made up with or without a reducing agent and the after treatment being the usual one. (B.P. 334,907 *idem*.) A new method of synthesising an indigo dye on the fibre is by steaming an arylthioglycol-o-carboxylic acid amide of the formula



where R is an aromatic residue which may be substituted (B.P. 299,069 *idem*).

Intermediates for Indigo

Recent patents for synthesising arylthioglycolic acids and other intermediates for indigos include B.P. 305,140 (*idem*) where o-thiocyano nitriles are converted to thioglycolic acids as follows:—

(a) Hydrolysis with H_2SO_4 to $Ar(CONH_2).S.CONH_2$.

(b) Reduction to $Ar(CONH_2).SH$ and finally condensation with chloracetic acid where $Ar=ar.(NH_2).SO_3H$ (1 : 2) is converted by the Sandmeyer reaction, followed by treatment with PCl_5 to $Ar(CN).SO_2Cl$ (b) then reduction and condensation with chloracetic acid gives $Ar(CONH_2).S.CH_2.COOH$. The reaction is illustrated with several substituted aryl derivatives.

Further, in B.P. 295,694 (*idem*) we have $Ar.(CN).S.CH_2.COOH$ (1 : 2) with alkaline hydrogen peroxide gives $Ar(CONH_2).S.CH_2.COOH$ and then treatment with caustic soda at $95^\circ C$. gives the thioindoxyl. Included is the production of 6-ethoxythioindoxyl. In B.P. 299,327 (*idem*) an arylamine is thiocyanated in the p-position by the method of B.P. 257,619 (*idem*) then hydrolysed to the corresponding mercaptan, condensed with chloracetic acid and the amine replaced with halogen by the Sandmeyer method. In B.P. 310,259 (*idem*) aromatic sulphonic acids are used for the hydrolysis of o-cyanothioglycolic acids to the carboxylamide derivatives.

Peroxide Bleaching

Some Factors which Influence Procedure

THE advantages of bleaching textiles with hydrogen peroxide has been discussed in a recent issue of our contemporary *The American Dyestuff Reporter*. Referring to the simplicity of this process, D. Stuart Quern, the author, states that there are, however, certain factors and requirements which bear decided influence upon the methods and procedure by which peroxide is used, and that the time element, expense of operation, and the condition of bleached cloth as regards absorbency and satisfactory dyeing qualities are all matters to be considered in relation to the process.

This accounts for the greatly increased practice of preparing goods for the hydrogen peroxide-silicate bath by means of a preliminary alkaline treatment or boil-off, or desizing, or both, proceeding on the principle that well prepared goods will be well bleached goods, regardless of the oxidising agent, and in addition will be in better condition for dyeing where this is necessary. Preparing goods well before the peroxide bath has been a decided factor in the wide expansion of peroxide bleaching. This is particularly true because the total expense of chemicals necessary is considerably less where preliminary treatment has preceded the peroxide bleach. In speaking of preliminary boil-off before peroxide bleaching, however, it is well to be more explicit about this. In some cases, it may be found advisable particularly where goods are very dirty, to submit the goods to a pressure boil-off in caustic soda, while in other cases, short open kier boils are sufficient. Regardless of what treatment goods may receive before the peroxide bleaching operation, it must be emphasised again that once the goods leave the kier, the entire procedure has been completed.

Dyeing Qualities of Peroxide-Bleached Cloth

Dyeing of bleached goods is a most important matter, and it is natural to inquire into the dyeing qualities of peroxide bleached cloth. The dyer wants the goods to reach him from the bleach house with a good white, of course, but he is primarily interested in cloth that is not going to show resists or take an excess of dyestuff to obtain his shades. Well prepared goods are essential to him in connection with his peroxide-bleached cloth. Many comparative tests as to the relative dyeing qualities of cloth bleached by different peroxide methods have been made in plants using this bleach. While the bleach would be unquestioned in every case, the best results would be obtained where the goods first received the caustic boil-off before the peroxide bleach. Peroxide bleaching of coloured goods, such as shirtings, towels, etc., of goods is also favoured, because of the simplicity by which such goods can be handled, and also because of the maximum of safety it offers for the colours. Bleaching of coloured goods may be accomplished in very much the same manner as has been described for white goods, but any pre-treatment given must necessarily be of a modified character. Procedure here differs somewhat in the application or extent of such preliminary treatments, but in

most plants some preliminary treatment for coloured goods is looked upon as best practice.

Simplicity of the Bleaching Process

In practice the goods are wet out and loaded into the kier, submitted to a bleaching solution of hydrogen peroxide and silicate of soda for the required number of hours, and finally pulled out through washers into the white bins. The temperature used for a hydrogen peroxide bleach bath ranges from a minimum of $180^\circ F$. to boiling point, with the higher temperatures favoured for attaining the maximum in results. Under these conditions the hydrogen peroxide process is completed in the kier, meaning, of course, that it is not necessary to pull cloth around the bleach house before reaching the white bins. This feature may well be considered worthy of consideration in the bleaching of all materials, and particularly important to the bleachery processing light fabrics where the least handling possible is desirable. The bleachery wants to turn out a piece of cloth of this type, not only just without damage, but in as perfect condition as possible, and peroxide offers considerable advantage in obviating the necessity of pulling light fabrics over a number of machines. Crooked fillings may often result from much pulling around of such fabrics, and this is a problem most easily solved by peroxide bleaching by reason of the lesser handling involved.

A certain small proportion of peroxide bleaching is done in wooden kiers, fitted with pump and heater. Such equipment is usually found in plants where the bleach output is comparatively small, and is not used to any great extent by those plants doing peroxide bleaching on a quantity basis. Theoretically, wooden kiers offer utmost freedom from catalytic action on the bleaching chemicals, but this supposed advantage will be found by actual comparative tests to be so negligible as not to be worthy of consideration where the iron kier equipment is kept in anything like ordinary good condition.

Scottish Dyestuffs Market

DURING August business conditions in Scotland were rather similar to those of the previous months, that is, they were on the whole rather depressed. There were, however, spurts of activity in one or two directions and dyestuffs sales were up as regards vat dyes and blacks.

The dyeing industry in Scotland has lost one of its outstanding figures on the death of Mr. Alexander Kennedy, of Anniesland, Glasgow, who founded the Castlebank Dye Works more than 50 years ago. One of his sons remains as managing director of the firm, while another is a director.

At Hawick, business has been a little better in the tweed trade and repeat orders for the winter season of 1931-32 have been coming in, and with the end of the holidays there should be a slight improvement in the general level of employment. The recent uncertainty in Germany is stated to have not been without effect in orders for the European market, but the effects of this should soon disappear. Woollen yarn dyers are still rather poorly employed.

It is understood that a new wages agreement in connection with the dyeing and cleaning sections of the trade is under discussion among the employees at the present time.

Manufacture of Dull Lustre Viscose Rayon

It has been proposed to incorporate various fats and oils in the viscose solution before spinning into threads so that the lustre of the resulting rayon shall be considerably less than normal. But a viscose solution is highly caustic alkaline and during the period which necessarily elapses before spinning saponification of the added fats occurs gradually and a sodium soap and glycerine are formed. It is now claimed (E.P. 344,288) that dull lustre viscose rayon can be similarly produced by adding a difficultly saponifiable ester of a hydrogenated phenol and a mono- or poly-carboxylic acid to the viscose solution instead of a fat or oil. The advantage thereby gained is that such a substance resists saponification and no serious change in the viscose solution occurs even when considerable time elapses between the addition and the spinning operation. The amount of ester employed need only be small, say, 1 per cent, or less calculated on the viscose. Methyl cyclohexanol esters of cinnamic methyladipic, stearic, and oleic acids are suitable.

Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.

Dyestuff Imports

ALTHOUGH dyestuffs production in this country has steadily developed under the Dyestuffs Act, we still, under our licensing system, import a considerable quantity of foreign dyestuffs that come to a substantial sum in the year. In the year 1930, for example, the total quantity of dyestuffs imported was 5,386,078 lb. of the value of £1,084,039. The corresponding figures for the year 1929 were 5,392,695 lb. and £1,097,916. Much the largest item in last year's list was the expensive vat colours, the quantity being 1,188,541 lb. and the value £206,341. In point of value the chrome and mordant colours come second with £220,346, and the other most important items are direct cotton colours, £171,251, intermediates £154,848, acid wool colours £105,030, lake colours £51,132, and basis colours £48,984. The cellulose acetate colours silk imported were of the value of £16,643. All these colours were, of course, imported under licence. The protective effect of the licensing system, however, is best realised from the number of refusals to grant licences. During 1930 the refusals covered a total of 194,807 lb., of the value of £37,091. In the previous year the refusals represented a value of £63,146.

New British Colours

An unusually large group of new British colours has just been put on the market by Imperial Chemical Industries. These include Caledon Black NBS, Solochrome Brown RGS and EBS, Lissamine CF, Tan Brown CF and Nigger Brown CF, Disulphine Blue FGS, and seven new Monolite colours—Bordeaux BLS Paste and Powder, Rubine BNS Paste and Powder, and Rubine GS Powder, Red 2BS Paste, Black BS Powder, Yellow GRS Paste and Powder, and Fast Yellow 10 GS Paste and Lumps.

Caledon Black NBS is described as excellently suitable for dyeing all types of cotton, linen and viscose silk yarns in the open beck and full vat black shades may be obtained at a reasonable cost. It is also suitable for chain warp dyeing in machines, but where vat blacks have to be dyed in circulating machines Caledon Black 2BMS is specially recommended.

Disulphine Blue FGS, a new acid colour, gives pure blue shades of very good fastness to stoving, decatizing, carbonising and acids, and as it possesses excellent dyeing properties it is of special value in piece dyeing either as a self shade or as a component of light mode shades. Its excellent levelling power makes it eminently suitable as a shading colour. This colour is of interest for dyeing wool and fur felt hat bodies, and is extremely valuable for shading purposes, giving excellent penetration. It dyes the wool or silk in union materials from an acid dyebath and leaves cotton, viscose, and acetate silk unstained. Owing to its brilliant shade and very good levelling power, Disulphine Blue FGS is of special interest for dyeing natural silk, giving shades of fair fastness to water, washing, and light. It is of interest to printers as a ground shade for the production of discharge effects and as an illuminating colour when used with stannous chloride discharges.

Two Solochrome Dyes

Of the two Solochrome colours, Brown RGS is of interest for all branches of wool dyeing and gives bright reddish

brown shades of very good fastness to light and milling. Its fastness to light and its good level dyeing properties make it specially suitable for the dyeing of piece goods; whilst its exceptionally good solubility makes it particularly suitable for use in all types of dyeing machines. It is soluble in acid dyebaths. It may be used, in combination with other chrome colours, for the production of mode shades, as it dyes equally well by all the three processes—i.e., afterchrome, chromate and chrome-mordant.

The Brown EBS is in powder form and gives deep nigger brown shades when dyed by either the chromate, afterchrome, or chrome mordant processes. The dyebaths are stable in the presence of acids. Materials dyed with this product possess excellent fastness to washing, perspiration, decatizing, acids and alkalis, and very good fastness to milling. It is of special value for the dyeing of loose wool and slubbing, in light and full shades, particularly in cases where a high standard of fastness to light and milling is required. It is also of considerable interest for piece dyeing. It is suitable for application to natural silk either by the after-chrome or chromium chloride mordant processes, giving shades of good fastness to washing. It gives full reddish brown shades on chrome tanned leathers when dyed with the addition of formic acid.

New Lissamine Colours

The three new Lissamine colours, Navy CF, Tan Brown CF, and Nigger Brown CF, have been produced in response to the urgent demand for a series of dyestuffs capable of covering fades and producing level shades on well worn garments. While in the manufacture of these dyestuffs the main object has been the covering of fades, the further important feature of good general fastness properties has not been lost sight of. The dyestuffs are applied in the usual manner from a dyebath containing sulphuric acid and Glauber's salt, and in this connection it should be noted that the covering of fades is accomplished most efficiently when dyeing is conducted in a strongly acid bath. Recommended proportions are as follows: Sulphuric acid 5 per cent., Glauber's salt 10 per cent. All three products leave cotton stitching practically unstained, and it is recommended that the following Chlorazol colours be used for filling the cotton, the operation being conducted in a fresh, cold bath: Chlorazol Brown 2G, Chlorazol Brown LFS, Chlorazol Black JHS, Chlorazol Burl Black ES.

The Monolite Colours

Of the batch of new Monolite colours, Bordeaux BLS Paste and Powder represent a standard bordeaux or maroon shade, intermediate between the bluish carmine and violet shades. It will find many useful outlets where a rich bordeaux shade of good fastness to light, oil, and water is demanded. The chief outlets will be for the manufacture of printing inks, and as a colouring medium for the wallpaper and paper surfacing trades. It is well adapted for the colouring of linoleum and for the manufacture of oil and water paint colours.

Rubine BNS is of particular interest to printing ink manufacturers on account of the dichroic character of the shades and beautiful clean bluish undertones. It is partially soluble in water and gives the most pleasing

results as a printing ink colour when precipitated as a calcium lake with calcium chloride. Lakes possessing improved light fastness, but rather less blue undertones, can be obtained by precipitating with strontium chloride.

Rubine GS Powder is the yellowest of the Monolite Rubines and produces a brilliant scarlet when struck down as a barium lake with barium chloride. A more pleasing bluish red shade of improved light fastness is obtained when the dyestuff is converted into the calcium lake. In general properties the colour is very similar to Monolite Rubine BNS and will find extensive use as a printing ink colour, where the beautiful clean bluish undertones are shown to advantage. In addition, it will find use in the manufacture of colour lakes for oil and water paints, wall-paper and paper surfacing colours, and for the colouring of linoleum.

The Red 2BS Paste belongs to the Rubine class of Monolite colours and, although not so blue as Monolite Rubine BNS, it produces brilliant bluish carmine shades of a dichroic character when precipitated as a calcium lake. The colour lakes produced with Monolite Red 2BS possess very good fastness properties and are eminently suitable for the manufacture of high grade printing inks of all classes, oil and water paints, and for the colouring of wall-papers and surface papers. In addition, it will find use for the colouring of linoleum and non-bleeding cellulose lacquers of good fastness to light. It is partially water soluble and forms lakes with the soluble salts of barium, calcium and strontium. The most pleasing shade is obtained when precipitated with calcium chloride, but the barium and strontium lakes are of interest in certain cases, although they do not possess the pure bluish undertone of the calcium lake.

The Black BS Powder is an insoluble pigment dyestuff possessing high grade fastness properties and is practically

inert to solvents. It is eminently suitable for cellulose varnishes where a high degree of covering power is not essential, and will find extensive use in the colouring of lacquers used in the manufacture of material where a surface coating of cellulose dope is applied.

The Yellow GRS Paste and Powder is a pigment yellow dyestuff redder in shade than Monolite Yellow GS, and possessing similar high grade fastness properties. It possesses a very high degree of fastness to light, oil and water and, therefore, comes into consideration as a substitute for Chrome Yellow, where the poisonous nature or the sensitiveness to alkali and atmospheric influences of this latter product precludes its use. It is used as an oil colour and as a colouring medium for high class wallpapers, surface papers, water paints and distempers fast to light and lime. In addition, it is employed for the production of artists' colours and high class printing inks fast to varnishing, and is particularly suitable for three-colour process work. Owing to its particularly good alkali fastness, it is well adapted for inks for soap wrapper printing and for exhibition carton-boards where the ink has to withstand the influence of light and very often the presence of alkali in the paper compound.

The Fast Yellow 10GS Paste and Lumps is in shade decidedly greener than Monolite Yellow GS, and possesses a pure greenish yellow tone unsurpassed for brilliance by any yellow pigment on the market. It possesses a very high degree of fastness to light, oil and water and, therefore, comes into consideration as a substitute for Chrome Lemon Yellow, where the poisonous nature or sensitiveness to alkali and atmospheric influences of this latter product precludes its use. Its uses and characteristics are much the same as Yellow GRS.

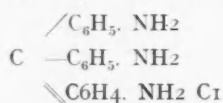
Basic Dyes and Intermediates: Recent Technical Progress

By L. J. Hooley

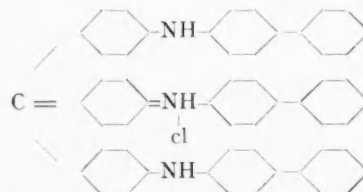
ALTHOUGH the basic dyes are now much less important than the vats and azo colours, they include such a wide range and present so much greater diversity in structure that such new work as is now published is always of interest.

New Aniline Blues

W. Dilthey's latest investigations on colour theory are in the Aniline Blue class. With R. Dinklage (*J. für prak. Ch.* 129, 1931, 214-30) he has synthesised a number of phenyl substituted dyes and examined their tinctorial properties. It will be remembered that while triaminotriphenylmethane hydrochloride, Rosaniline base, is red (constitution, quinonoid form, shown below), substitution of the amino groups



by alkali groups gives increasing depth of colour through the Methyl Violets to Iodine Green, this forming one of the classical examples of Nietski's rule that colour is deepened by adding substituents to increase the molecular weight. If the substituting groups are phenyl groups so that we have three $\text{—C}_6\text{H}_4\text{.NH. C}_6\text{H}_5$ chains attached to the central carbon atom the colour is blue and we get the Aniline Blues which differ from the simpler derivatives in being insoluble in water and which are used as spirit blues. Dilthey and Dinklage have extended this process of substitution by using the chain $\text{—C}_6\text{H}_4\text{.NH. C}_6\text{H}_4\text{. C}_6\text{H}_5$ and also the corresponding chain in which the last phenyl group is attached through O or S and products such as that annexed are obtained.



They found, however, that these three additional phenyl groups had a negligible effect on the colour, all the new products being blue. Dilthey uses the carbonium formula, and in this the chlorine atom is not attached to the nitrogen. With this formula, the additional groups may be considered to be too far from the colour source to exert any influence. As regards the other properties such as fastness and solubility the new compounds are of little interest.

New Triaryl Methane Colours

Also of interest in connection with the question of constitution of the triaryl methane colours is the work of L. C. Anderson (*J. Amer. Chem. Soc.*, 52, 1930, 4567). He describes the absorption curves of a number of triphenylmethane derivatives, i.e., triphenylcarbinol, triphenylmethyl chloride, sulphate and perchlorate in sulphuric acid and other solvents. From the general similarity of the curves the compounds are believed to be present in quinonoid form.

Among new triaryl methane compounds, there may be mentioned violet dyes obtained by condensing *m*-2-xylylene with a diaralkyldialkyldiaminobenzhydryldisulphonic acid and oxidising. The production of the hydrol from the triarylmethanedisulphonic acid the condensation with the

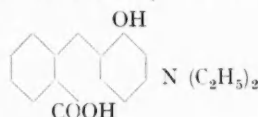
xylydine and the oxidation of the leuco compound can be carried out together in the xylydine. (I.C.I., E. H. Rodd, and L. Sharp, B.P. 334,689). In B.P. 338,126 (I.G.F.A.-G) *o*-hydroxylated di- and tri-arylmethanes are brominated or chlorinated.

Lake Colours

In lake colours the following recent patents may be noticed. According to B.P. 318,834 (I.G.F.A.-G) the presence of soluble sulphonic acids is an advantage in the precipitation of basic colours from aqueous solution. Under these circumstances precipitation takes place smoothly and readily and the products obtained can be readily filtered. Being in a fine state of division they are suitable for carbon papers and typewriter ribbons. Among the sulphonic acids mentioned are dibutyl-naphthalene sulphonic acid and nuclear substituted anthracene sulphonic acids and their hydrogenated products. In an example, aluminium sulphate and sodium carbonate are used to precipitate alumina and the well washed product is stirred up with a solution of crystal violet and the lake precipitated with dibutyl-naphthalene sulphonic acid.

H. Clayton in *J. Soc. Dyer and Col.*, 46, 1930, 154, gives a general description of the manufacture and use of colour lakes, including vat colours as well and their application in a great variety of industries including paint, enamel, rubber, lacquer, linoleum and leather and paper.

Aminoxylol ($\text{OH} : \text{NH}_2 : \text{CH}_2 : \text{CH}_3 = 1 : 3 : 2 : 4$) will condense with a 4'-di-*o* mono-alkylamino-2'-hydroxy-2-benzoylbenzoic acid to give products such as that shown below, which after the necessary esterification of the free



carboxyl group are very bright bluish red Rhodamine dyes which are suitable for use in making complex pigments with phosphotungstic and similar acids (I.C.I., and M. Wyler).

The parent substances of a new series of sulphone phthaleins having halogen in the sulphobenzoyl anhydride residue are described in *J. Amer. Chem. Soc.*, 52, 1930, 4954, by W. C. Boyd and A. W. Rowe, together with methods of making the phthaleins themselves.

Acridine Derivatives

Therapeutic agents with valuable bactericidal properties are obtained in the acridine series by making double salts of 3:6-diamino-10-alkylacridinium bases and 3:6-diamino acridine. A variety of ways of combining the two acridine components are described and claimed. As an example the methylacridinium chloride and the acridine hydrochloride are treated together in water at 80° C. A feature of the new compounds is their much greater solubility in water and another advantage is that they show constant composition; the double compounds obtained in the example above has a solubility of 15%, while that of each of the starting materials is only about 1% (B.P. 328,212, I.G.F.A.-G).

Synthetic Tanning Agents

The use of synthetic agents, e.g., sulphurised derivatives of phenols and formaldehyde naphthalene sulphonic acid condensation products, should be extended by the discovery of a satisfactory discharge method. The usual method of obtaining tannin discharge styles, namely with caustic soda, fails with tannin substitutes as it does not sufficiently remove the substitute from the fibre. In the new process the tannin mordanted material is treated with an oxidising agent, preferably a chlorate and then dyed with the basic colour. A suitable discharge paste for use with a sulphurised *o*-chlorophenol is a mixture of British gum, water and sodium chlorate with potassium ferricyanide, ammonium citrate, dimethyl-phenylbenzyl ammonium chloride and glycerine. The process gives white discharges fully equal to those obtained with tannin itself (B.P. 325,562, I.G.F.A.-G).

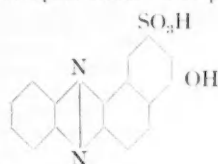
Almost simultaneously the problem of reserving, as distinct from discharging, sulphurised phenols has been solved (B.P. 325,935, I.C.I. and L. Smith). In this a metallic halide, especially zinc chloride, but excluding an alkali metal halide is applied to the fabric in for example a paste containing

British gum and China clay. After drying, padding with the mordant and again drying and acidulating, the fabric is dyed with the basic colour and the white thus cleared. The reserves may be obtained instead of white by appropriate devices.

As regards the synthesis of sulphurised phenol tannin substitutes Sandoz (B.P. 325,388) describes a new technique, this consisting in boiling the phenol under reflux with water, lime and sulphur. The products have greater fixing power than some similar previously described phenol products. They are obtained in the first instance as calcium salts and can be converted to the soluble ones by usual base exchange treatments.

Azine Derivatives

A number of miscellaneous products have been described. Azo derivatives of azines are obtained by coupling 4-hydroxy-2-sulpho- α -naphthaphenazine. The products are acid wool



colours and may also be mordanted. As an example *p*-nitraniline gives a red (B.P. 325,896, S.C.I.B.). A 1:8-naphthasultam carrying an arylamino substituent in the 3-position and preferably also a sulpho group in the 6-position can be condensed with a *p*-nitroso-dimethylaniline or oxidised with a *p*-diamino to give a phenonaphthazine (U.S.P., 1,723,199, W. Neelmeier, T. Nocken and Gen. Aniline Works).

Although the monosubstituted azines, eurhodols and eurhodins including hydroxy and aminoazines are not generally of technical value, the amino-naphthaphenazines and certain derived compounds from *o*-phenylene diamines and 8-acetyl-amino-1:2-naphthaquinone sulphonic acids give fast yellow dyes distinguished by brightness and fastness to light.

8-Acetylamino-1:2-naphthaquinone-5-sulphonic acid and *o*-phenylenediamine condense to 8-acetylamino-1:2-naphthaphenazine-5-sulphonate which dyes wool in clear greenish yellow shades. After removal of the acetyl group by boiling with dilute sulphonic acid the corresponding shades are orange red. Benzoylation then gives a reddish yellow. If desired the sulphonic acid group may also be removed by treating with mineral acids under stronger conditions than those employed for removing the acetyl group (B.P. 328,291, I.G.F.A.-G). While speaking of hydrolysis of azinesulphonic acids, B.P. 322,029 may be referred to, where it is shown that in the desulphonation of 1:2-naphthaphenazines or 1:2:2':1' dinaphthazine by caustic alkali previously carried out either in the alkali itself or in 36% solution, a strength of 10% is actually sufficient.

The Grasselli Medallist

The Grasselli Medal, American industry's annual tribute to distinction in applied chemistry, has been awarded for 1931 to Dr. L. V. Redman, of Bloomfield, New Jersey, vice-president and director of research to the Bakelite Corporation. The selection of Dr. Redman, the new president of the American Chemical Society, was based upon industrial studies, the results of which were embodied in a paper on "Cost of Research and its Apportionment" read before a joint meeting of the American Section of the Society of Chemical Industry with the New York Section of the American Chemical Society, the Societe de Chimie Industrielle, and the American Electrochemical Society on December 9, 1927. The medal will be bestowed upon Dr. Redman at an assembly of the chemical societies of the New York area on November 6. Professor D. D. Jackson, executive officer of the Department of Chemical Engineering, Columbia University, will make the speech of presentation. Mr. F. W. Willard, works manager of the Western Electric Co., will describe the work of the medallist. Dr. Redman will deliver an address on "Research as a Fixed Charge."

The Future of Rayon

Displacement of Natural Fibres Anticipated

In the following article the writer takes the view that rayon has now advanced just as far as its properties will allow it. It has many obvious advantages over the older fibres, yet it has properties which reduce its value. Research will show how these faults can be eliminated; ultimately rayon should largely displace its natural competitors.

THE value of a raw material for manufacturing purposes must be directly dependent on its present properties and also on the extent to which such properties are likely to be improved in the future. This applies to rayon no less than to other materials which can be converted by manufacturing processes into articles of utility; but it must of course be noted that rayon has particularly to meet competition from the older established fibres. It must therefore be asked how far the present properties of rayon allow it to compete with and displace such fibres as cotton and wool, and how far might it be reasonably expected that these properties can be improved so as to render the competition of the older fibres smaller and ineffective.

In the first place it is a great advantage that rayon can be directly manufactured in the form of a yarn which is suitable for weaving or knitting. It is not necessary, as in the case of raw cotton or wool, that the fibres should be graded, freed from particles of dirt, repeatedly straightened by combing and similar operations, spun into yarns with the assistance of drafting processes, then gassed, and bleached by tedious chemical processes. A rayon yarn is produced in the form in which it is wanted by fabric manufacturers and it requires no bleaching treatment. Thus in comparing the prices of cotton and wool with rayon it is necessary to consider the price, not of raw cotton or wool, but of the cotton and wool yarns; if this is done then the price of rayon is comparatively cheap.

A Wide Range of Properties

Turning now to the properties of rayon it is necessary to remember that three types of rayon are now available and since these have different properties it is possible to select the rayon to suit the class of material being manufactured. If extreme softness is required then a fabric should be made from a cuprammonium or cellulose acetate rayon; if the maximum durability to all treatments is desired then viscose rayon will be found suitable; and if unique dyeing properties are necessary then cellulose acetate rayon must be used. Staple rayon yarns (composed of short rayon fibres) are serviceable in the manufacture of fabrics having a wool-like and warm handle. The use of coarse (high denier) yarns enables cheap goods to be produced but having a somewhat firm handle, whilst extremely soft materials can only be manufactured from the lowest denier yarns which are somewhat expensive. Thus a wide range of properties can be secured in manufactured rayon goods merely by variation in the type and fineness of the rayon yarns used.

The buying public appears to exercise great discrimination as regards the lustre of the rayon goods which it purchases. It is not sufficient that a garment or fabric has an attractive sheen; it must have a sheen which is definite but unobtrusive. For this reason many rayon manufacturers market their yarns in two types—bright and dull lustre. Also dyers and finishers of rayon goods are able by special processes to give them a matt or almost lustreless appearance. Under such conditions the lustre of a rayon material can be produced with any desired degree of lustre. Softness or "kindliness" of handle is also desired in the majority of rayon materials and although this property is mainly decided by the rayon manufacturer it can be assisted by the finisher and also by the dyer. Much research has indicated that a number of aromatic substances have the power to render a rayon smoother and softer to the touch: these are now coming into general use.

The dyeing of rayon is not now nearly so difficult as it formerly was. Both manufacturers of rayon and dye makers have contributed to this improvement. Dye makers have succeeded in producing more even-dyeing colours whilst rayon producers have perfected their methods such that the quality of their rayon is now more uniform.

Existing Defects as Textile Material

These considerations show that rayon is a very serviceable textile material in that it readily allows the production of fabrics of numerous kinds having various properties; rayon can be readily adapted to suit the demands of the public as regards lustre, softness and colour. In this respect rayon is superior to other fibres. But at the moment, rayon is not perfect; it has faults and it lacks certain properties which are desirable. Firstly, all rayons suffer a temporary loss of strength when wetted and in this they are much inferior to all other fibres such as cotton, wool and silk. Even the so-called "strong" rayons of the Lilienfeld type suffer from this defect. Another serious defect is that the properties of rayon are much too easily affected by the stretching to which it is subject. Stretching increases the liability of a rayon to crease; it increases its lustre and affects its absorption of dyes during a dyeing process. Also rayon is too susceptible to moisture; it stretches so much more easily when damp than knitting and weaving faults can often be traced to this factor.

With all its faults, rayon has secured for itself a most important position among textile fibres. During the past few years its production has increased rapidly, and rayon finds a place in a very large number of cotton, wool and silk materials. For many types of cotton fabric the presence of rayon is essential. And now it has to be noted that the production of rayon is not expanding appreciably; it appears to have permeated the textile industry to its maximum extent. It is believed that its present stationary position is due to limitations in the properties of rayon itself. With its present advantages and disadvantages rayon has entered into the textile industry—into the company of cotton, wool and silk and "rubbed shoulders" with these. By reason of its advantages it has displaced these older fibres to a certain extent but because of its disadvantages it can displace them but little further. The consumption of rayon must in future be governed by the general world demand for textile materials if rayon remains as it is to-day.

Suggested Avenues for Future Improvements

Rayon is a synthetic fibre whose properties can be regulated by conditions of manufacture; its properties are not subject to such little-understood forces as those which naturally operate in the formation of cotton or wool. By research it should be possible to improve rayon; such improvement is thus merely a matter of labour and time. Our present knowledge of the industry would suggest that improvements will come along three main avenues. In the first place it is clear that the raw cellulose from which a rayon is manufactured has a large part in determining the properties of the rayon itself. Recent X-ray investigations of rayon and cellulose materials have shown that the properties of fibres are largely determined by the size and internal arrangement of the micellae and that these can in turn be governed by the raw cellulose used in the rayon manufacture. At the present time almost all rayon is made from cotton or wood pulp or a mixture of these. But there is no reason whatever why research should not reveal better raw materials which could result in improved rayons. It may well be that within some plant, tree, or other form of vegetable growth there is awaiting exploitation a type of cellulose which is capable of giving exceedingly valuable rayon.

Progress can also come through improvement in manufacturing processes. Rayon manufacture merely consists of converting loose cellulose into continuous lengths of cellulose fibre; the complicated processes of preparing cellulose solutions whether by the viscose, acetate, or cuprammonium methods are just means for affecting this conversion of change of form. No one could deny the possibility that new methods of conversion may be discovered and that by use of these an improved rayon be produced.

Dyestuffs at the Faraday Exhibition

Historical Developments in Dyes and Dyeing

FROM the discovery of benzene by Faraday (1825) to the latest commercial dyestuffs is a long one brilliant stage in the progress of chemistry. During those years succeeding generations of chemists here produced dyes of every hue for every kind of textile material. In tracing the history of these colours at the recent Faraday Exhibition, it was a happy idea of Professor H. E. Armstrong to show brilliant hues on delicate fabrics instead of exhibiting mere samples in uninspiring bottles. In all, more than one hundred different dyestuffs were assembled by the generous assistance of a number of colour-making and textile firms, and the stand shown in the accompanying illustration was actually presided over by Professor Armstrong, who was in later years a colleague of Sir William Perkin, whose famous discovery of Mauve was only made possible by Faraday's earlier discovery of benzene.

The First Synthetic Dyestuff

The first synthetic dyestuff (Perkin's Mauve) was discovered, with premeditation, by William Henry Perkin, during the vacation working in his rough laboratory at home.



DYESTUFFS STAND AT FARADAY EXHIBITION.

He was seeking to explain an observation made with an aniline derivative and resorted to the cheaper aniline. Mixing solutions of aniline sulphate and potassium bichromate, he obtained a black precipitate; he soon found that this contained a colouring matter, with the properties of a dyestuff. So practical was his outlook that, having patented the dyestuff, on August 26, 1856, he decided to undertake its manufacture, and with the aid of his father and brother established a works at Greenford Green, near Harrow.

Others naturally followed in his footsteps. In 1859 the French chemist, Verguin, treating aniline more drastically, discovered Fuchsine, also called Magenta and Roseine, finally Rosaniline by Hofmann. This soon became a serious rival to Mauve. It was first manufactured successfully here by Nicholson, of Simpson, Maule & Nicholson. Beautiful as was this dyestuff, it was soon converted into even more beautiful derivatives, blues, violets and even a green. The value of the blue was enhanced by Nicholson, who made it soluble in water, by adopting the sulphuric acid treatment applied by Faraday to naphthalene and by Berzelius to

indigo. New dyestuffs then came forward sporadically, especially from Manchester, where Grace Calvert and Roberts, Dale & Co., were active, the latter being supported by two skilful German chemists, Caro and Martius. Caro made a yellow from aniline, Martius a brown from diamidobenzene; Martius also made the first technical use of naphthalene by introducing a yellow dinitronaphthol, obtained from naphthylamine and nitric acid. Calvert produced all but pure carboic acid on a large scale, and from this picric acid. He also developed the use of Aniline Black, formed upon the fibre.

Discovery of Alizarin

The dyestuffs industry became more fully developed on the discovery by Graebe and Liebermann, in 1868, when Alizarin, the dyestuff from madder-root, next in importance to indigo, was derived from the coal tar hydrocarbon Anthracene, $C_{14}H_{10}$, discovered by the French chemists, Dumas and Laurent. They then obtained alizarin by oxidising anthracene to anthraquinone, brominating this and heating the dibromo-derivative with caustic potash. On learning of Graebe and Liebermann's success, having some anthraquinone in his possession, Perkin set to work and soon found an easier method—involving treatment of anthraquinone with sulphuric acid and subsequent fusion of the sulphuric acid with alkali. The Germans were at work upon similar lines and were ahead of him by a day in taking out a patent for the process (1869). Nevertheless, so practical was he, that he was the first in the field as a manufacturer.

The aniline dyestuffs thus far used all produced their own colours upon fabrics and were primarily applicable only to silk and wool. Alizarin and the related compounds that were soon obtained were not used as such, but together with mordants, on wool and cotton, giving rise to reds when used with alumina and tin mordants and to purples with iron and chromium. The industry really became revolutionised when the manufacture of Azo-dyestuffs was first put upon a considered scientific basis.

Azo-Dyestuffs

The diazo-reaction was discovered by a German student, Peter Griess, in Marburg (1850). Shortly afterwards he came over here as assistant to Hofmann, and in 1862 became chemist in Allsopp's Brewery. His discovery was that it was possible to substitute a single atom of nitrogen for three atoms of hydrogen in an ammonium salt. The products were very unstable but also very reactive, particularly towards amines such as aniline and phenols, giving rise to stable amino-azo and hydroxy-azo compounds, all of which were coloured, and dyestuffs. Working here, with Williams, Thomas and Dower, Dr. Otto Witt, in the middle seventies, guided by theoretical considerations as to the relationship between chemical structure and dyeing properties, he systematised the use of Griess's method. He also effected a great technical improvement by using sodium nitrate in conjunction with an acid, so as to liberate nitrous acid, a very unstable acid, at the required moment. He produced Chrysoidine, intermediate between the yellow and the brown made by Caro and Martius, in Manchester, obtaining a dyestuff which was not so fugitive as Caro's product, and of a more desirable colour than Bismarck brown.

The possibilities opened out were innumerable; workers everywhere took up the search. Only yellow azo-colours had been made. Reds were soon produced, especially when the naphthols and naphthylamines were used. Then came the all important discovery, in 1884, by Böttiger, of Congo-red, a direct cotton dye. Previous azo-dyestuffs had required a mordant. This was made from benzidine. Very superior dyestuffs were soon obtained by using related compounds and the range of colour increased. Actually such dyestuffs were diazo-compounds; by rediazotising and coupling again trisazo and even tetrakisazo compounds were obtained, giving every shade of colour up to almost black. Great improvements were also effected in dyeing, as it became possible to produce some dyestuffs with advantage upon the fibre. A

particularly remarkable instance is that afforded by the use of β -naphthoic anilide, $C_{10}H_6(OH).CO(NH.Ph)$.

Triphenylmethane

The structure of the Rosanilines remained a riddle up to 1870, when Emil and Otto Fischer were able to show that it was a triamidotriphenylmethane, derived from the hydrocarbon $CH(C_6H_5)_3$. Perkins and others had at their disposal only a very impure benzene containing toluene, consequently the aniline that was used contained much para- and some ortho-toluidine. It was soon found that rosaniline was not to be obtained from aniline itself. When the Fischers' discovery was made, it became clear that the toluidine gave the necessary coupling carbon atom. Improved synthetic processes were soon devised for the preparation of a wide range of triphenylmethane dyestuffs. Noteworthy is the use made of the phosgene gas discovered by Sir H. Davy's brother in 1812. By the interaction of the chloride and amines such as dimethylaniline, Michler was able to prepare a series of ketones from which at a later date many new rosanilines were made.

Indigotin and Indigoid Dyes

Nothing would have surprised and delighted Faraday more than to find that Indigotin, the colouring matter of Indigo, the oldest and most widely used of natural dyestuffs, was a derivative of benzene, to be made artificially from the hydrocarbon, through aniline, on any desired scale and cheaper than natural indigo. It cost Baeyer twenty years of intense labour to establish the formula of Indigotin and to produce the substance artificially. Being insoluble, Indigotin cannot be used directly: it must first be converted in the dye vat into indigo-white, a soluble substance; the goods to be dyed, having been soaked in the solution of indigo-white, are exposed to the air, when the two atoms of hydrogen added to the indigotin during reduction are removed and the pigment reconstituted within the fibre. In recent years, a great variety of indigoid dyes, resembling indigo in general structure and behaviour, have been produced, particularly in Switzerland, at Basle, under the direction of Dr. Engi. Among these is a tetrabromindigotin which has the advantage of being more stable than indigo. Thioindigo, containing sulphur in place of the nitrogen group (NH), is of particular interest on account of its red colour.

Foreign Contracts

Present Position under New Rates of Exchange

DIFFICULTY is being created by contractors abroad owing to the depreciation in the pound sterling. Some foreign firms who have made contracts to sell their goods to English houses on a sterling basis are showing a tendency to repudiate these contracts, and in certain cases they have gone so far as to assert that unless they are paid at the rate of the fallen value of the pound they will not deliver the goods.

The Gold Standard (Amendment) Act, according to a high authority at the Department of Overseas Trade, does not in any way invalidate contracts entered into at sterling prices. Buyers in this country are entitled to pay in sterling, and sellers in this country are entitled to payment in sterling. With regard to contracts at sterling prices in respect of which foreign suppliers refuse to deliver unless the buyer agrees to meet the difference in exchange, the suppliers should be held to their contracts, as the British buyers in many cases have already contracted to resell the goods on the basis of sterling and will be obliged to fulfil their own contracts. It should be borne in mind that in cases where contracts have been entered into in terms of the foreign currency instead of sterling, the British buyer will certainly have to honour the contract even though loss will be involved. This is a point which the foreigner has overlooked.

Where an attempt is made to demand payment at the depreciated rate, the British trader should report the full circumstances to his particular Chamber of Commerce, and there is no doubt that combined action will be taken by the Department of Overseas Trade and British Chambers of Commerce to ensure that the contracts are carried out in accordance with the agreements entered into.

Bradford Technical College

AT the Bradford Technical College the full-time day diploma courses in chemistry and dyeing and in metallurgy extend over a period of four years. If a student has obtained a school certificate on leaving the secondary school, he is exempted from the entrance examination, and if in addition he has obtained the matriculation examination of the University of London or obtained exemption from this, he may take up the course in chemistry or in chemistry and dyeing, which leads to the B.Sc. degree of the University of London or the Associateship of the Institute of Chemistry. There are certain engineering subjects in these courses which are compulsory in addition to mathematics, physics and chemistry. Advanced students help with many technical problems which are submitted to the College, and consequently are trained to view problems both economically and scientifically. In the textile and dyeing departments the workshops are fitted with machines similar to those actually employed in industry. In 1919-20 a new chemical laboratory for physical chemistry was added to the College, and again this year further laboratories are in course of construction. Facilities are given to those students who wish to continue for a period after obtaining a degree to carry out research work on either technical or pure scientific problems. The College is recognised by the Institute of Chemistry as a training centre for the Associateship, and is in close contact with industry, and many firms allow their apprentices to attend the College as part-time day students.

Constitution of Cyanidin Chloride

IN a communication to the *Journal of the Indian Chemical Society*, 1931, 8, 329, Dr. M. Nierenstein discusses the constitution of cyanidin chloride, a substance which is of interest both to chemists and to botanists on account of its relationship to the anthocyanidin pigments of flowers. Since cyanidin chloride breaks down on hydrolysis to phloroglucinol and protocatechuic acid, it is necessary to ascertain the mode in which the two nuclei of these products are linked in cyanidin. Willstätter ventured in 1914 to assign a definite structure to the latter compound, since it was supposed that it could be produced from the anthoxanthone quercetin by reduction. The fact that this reduction theory, though strongly favoured by Everest, Willstätter, and other chemists, appears to conflict with botanical evidence led Malkin and Nierenstein to reinvestigate the matter. These authors (*Jour. Amer. Chem. Soc.*, 1930, 52, 2864, were able to show that reduction of quercetin leads to a dimolecular product, quercetylene chloride, and not to the monomolecular pentahydroxyflavylium chloride, which has the structure assigned by Willstätter to cyanidin chloride. It appears that both quercetylene chloride and pentahydroxyflavylium chloride have been identified with natural cyanidin chloride, but the evidence on which identity of composition is based is stated to be inconclusive. Furthermore, X-ray analyses of the natural cyanidin chloride and the synthetic product have revealed their dissimilarity. Thus there appears to be some doubt about the actual structure of cyanidin chloride. A possible solution of the problem, based on the fact that Freudenberg has reduced the compound to epicatechin, is tentatively suggested in the paper.

Dyestuffs Licences for September

THE following statement relating to applications for licences under the Dyestuffs (Import Regulation) Act, 1920, made during September has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee:—

The total number of applications received during the month was 704, of which 613 were from merchants or importers. To these should be added 4 cases outstanding on August 31, making a total for the month of 708. These were dealt with as follows:—Granted, 660 (of which 654 were dealt with within 7 days of receipt); referred to British makers of similar products, 33 (of which 31 were dealt with within 7 days of receipt); outstanding on September 30, 15. Of the total of 708 applications received, 685 or 97 per cent., were dealt with within 7 days of receipt.

Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

Communications relating to editorial matter for the Dyestuffs Monthly Supplement should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.

End of U.S.A. Dye Census

In this country there has often been a demand for fuller particulars relating to the dyestuffs industry, and the Government attitude has been criticised in comparison with that of the United States, where a volume embodying the Tariff Commission's Census of Dyes and other synthetic organic chemicals has been published for some years. It is now announced that the publication of this interesting volume is to be discontinued. The ostensible reason is the need for economy, but another suggestion is that the information was of more advantage to foreign than to home producers, domestic manufacturers being divided on the advisability of publication. The original reason for the survey has disappeared. The United States Revenue Act of 1916 provided that duties then levied on dyes would be removed in five years if domestic production had not reached 60 per cent. of consumption, and President Wilson authorised the survey to ascertain the rate of progress in the industry. The United States has long since exceeded the 60 per cent. level.

The Dyestuffs Act

In view of the decisive result of the general election and the composition of Mr. MacDonald's new Cabinet, there should be very little doubt about the renewal of the Dyestuffs (Import Regulation) Act, when it runs out at the end of this year. Both Mr. W. Graham and Mr. Tom Shaw, members of the late Government, not only took up an unsympathetic attitude towards the Act, but treated the future of the dyestuffs industry with an unconcern that caused great disappointment and resentment in the industry. In the new Government, Mr. Walter Runciman will be at the Board of Trade, and as a business man will be able to appreciate the business side of the matter in a way that his predecessor quite failed to do. Mr. Runciman is, of course, a well-known Liberal, with Free Trade sympathies, but it is understood that his views incline to prohibition of excessive imports rather than to tariffs. He should have little difficulty, therefore, in understanding the principle of the Act, which, without taxing foreign dyes or prohibiting their entrance into this country, so regulates their importation as to afford practical protection of the home industry where it needs it. Fortunately, the services of Mr. Woolcock, who has been so closely associated with the administration of the Act from the outset, are still available for negotiation purposes, and it is confidently expected that the renewal of the Act will now be assured.

A British Standard Colour Card

It will be recalled that just a year ago the British Engineering Standards Association issued a British Standard Schedule of Colours for Ready Mixed Paints (B.S.S. No. 381-1930), containing 57 agreed colour patterns with self-explanatory colour names, and the colorimetric value of each colour in the appendices.

In response to a number of requests from those not requiring the scientific colorimetric analysis, for something of less bulk and cost, the Association has published a British Standard Colour Card which includes all the colours given in the schedule. Everybody interested in the choice of ready mixed paint will welcome this new publication.

Already it is foreseen that it will become the practice of the householder to give instructions to decorators simply

by specifying interior or exterior decoration by the number of the British Standard Colour for each particular purpose or room. Copies of the B.S. Standard Colour Card (Reference No. 381C-1931) may be obtained on application to the Publications Department, British Engineering Standards Association, 28, Victoria Street, London, S.W.1, price 2s. 2d. post free.

Two New Caledon Colours

In the steady output of new colours by Imperial Chemical Industries are two additions to the well-known and extensive range of Caledon dyestuffs, namely, Brilliant Purple 4RS and Direct Black ACS.

Caledon Brilliant Purple 4RS gives shades redder and slightly brighter than those produced by the older brand, Caledon Brilliant Purple 2RS, and is of considerable interest to dyers of cotton, linen and viscose. It is of the greatest interest to calico printers, to whom the brilliancy of shade and excellent printing properties will make a strong appeal, and is suitable for direct printing by the potash-Formosul method and for use as an illuminating colour in vat discharge printing (Formosul-Metabol process).

Caledon Direct Black ACS is an important addition to the range in that it provides a means of producing grey and black shades of excellent fastness, from an alkaline hydrosulphite bath without the necessity of the subsequent oxidation treatment usually associated with the dyeing of vat blacks. Caledon Direct Black ACS is therefore of great interest to dyers of cotton, linen and viscose yarns and to a lesser extent to cotton piece dyers for the production of greys and for shading purposes. It is eminently suitable for machine dyeing and for calico printing by the caustic soda-Formosul method.

New Chlorazol Colours

The new I.C.I. colours also include three additions to the range of Chlorazol dyestuffs.

Chlorazol Fast Grey 2BKS is one of the fastest to light direct cotton greys available, in addition to which it possesses very good general fastness properties. This colour is of interest for dyeing all forms of cotton materials and is specially suitable for the dyeing of cotton piece goods owing to its very good fastness to light, acids, alkalis and alkaline storage, whilst its excellent level dyeing properties further enhance its utility for this class of work. It is specially interesting for the production of light silver grey shades on cotton-viscose pieces. It possesses excellent solubility and is eminently suitable for the dyeing of cops, beam warps, etc., in the various types of dyeing machines. It possesses very good affinity for natural silk, in both the weighted and unweighted forms, when dyed from an acid dyebath. It gives attractive grey shades on these materials which possess the same high standard of general fastness as when dyed on cotton. Its affinity for tin-weighted silk makes it of particular interest for the dyeing of crepe materials. This colour is suitable for the direct printing of cotton and viscose and is dischargeable with Formosul giving good whites on cotton and natural silk and somewhat creamy whites on viscose. It is suitable for the dyeing of paper and will be of interest in those cases where fastness to light, acids, and alkalis is of importance.

Chlorazol Catechines G and GRS are particularly suitable for the production of yellowish brown or cutch shades on

cotton pieces, although at the same time they are suitable for application to all forms of cotton materials. These colours possess good fastness to water, alkalis, alkaline storage and hot pressing and are of fairly good fastness to acids. They possess good solubility and are quite suitable for machine dyeing. It is to be noted that the fastness to water, washing and light of these colours may be improved by an after treatment with copper-bichrome. Chlorazol Catechines G and GRS are suitable for the direct printing of cotton, viscose, wool and natural silk and will also be of interest as ground shades for the production of white discharge effects by the Formosul method. They are suitable for the dyeing of natural silk, viscose, paper, jute, straw, tagal and wood-chip and for application on full-chrome leathers.

A Monolite Green Paste

Finally the new list includes an addition to the Monolite series—Monolite Green 3BS paste and powder. This product is a green pigment dyestuff bluer and brighter in shade than Monolite Green BS and possessing similar high grade fastness properties. Possessing particularly good fastness to light and alkali it will be found eminently suitable as a colouring medium for wallpapers, paper surfacing colours, and distempers. In addition it is adapted for the production of fast to light printing inks, oil and water paints and artists' colours, and for the colouring of cement compositions where the fastness to alkali is an essential feature. The excellent fastness to light and caustic alkali renders this colour specially suitable for the production of inks for exhibition carton boards where the ink has to withstand the influence of light and very often the presence of alkali in the paper compound. Monolite Green 3BS is insoluble

in water and requires no precipitating agent. It is simply ground or mixed with the required substratum into the process medium.

A Durindone Late Scarlet

Durindone Lake Scarlet YS Powder, an addition to the range of I.C.I. pigment dyestuffs, is an insoluble yellow shade scarlet pigment dyestuff possessing high grade fastness properties. Possessing good light fastness it is particularly suitable for the production of high-class printing inks, oil and varnish paints, artists' colours, water paints and distempers. Its excellent fastness to caustic alkalis makes the colour specially suitable for the manufacture of inks for soap wrapper printing and for use wherever fastness to alkali is an essential property. It is insoluble in water and requires no precipitating agent.

Dyestuffs Licenses for October

The following statement relating to applications for licence under the Dyestuffs (Import Regulation) Act, 1920, made during October has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee:—

The total number of applications received during the month was 1,038, of which 909 were from merchants or importers. To these should be added 15 cases outstanding on September 30, making a total for the month of 1,053. These were dealt with as follows:—Granted, 1,004 (of which 992 were dealt with within seven days of receipt); referred to British makers of similar products, 42 (of which 31 were dealt with within seven days of receipt); outstanding on October 31, seven. Of the total of 1,053 applications received, 1,023 or 97 per cent. were dealt with within seven days of receipt.

Basic Intermediates for Dyestuffs: No. XLVI—Some Derivatives of Benzidine

By "Consultant"

THE preparation of the trio of aromatic diamines, benzidine, tolidine and dianisidine has been described in an earlier contribution to this series. Their derivatives are also of importance as dyestuffs intermediates; the halogenated benzidine derivatives are especially valuable since the presence of the halogen atoms in the benzidine nucleus tends to give dyes of a deeper and more definite blue shade than those from unsubstituted benzidine. This is especially true of the azo-dyes. On the other hand, it has been observed that the dyes obtained from nitrobenzidine have a distinct tendency towards the red end of the spectrum, redder shades being obtained than those from the analogous dyes in which the nitro-group is absent. As a case in point the so-called Anthracene Red may be cited; it is obtained by the diazotisation of nitrobenzidine and coupling with salicylic acid and then with naphthol-4-sulphonic acid. It is not, therefore, an anthracene compound, the name Anthracene Red being applied in order to indicate its fastness. Actually it is a good scarlet dye, whilst on the other hand the corresponding compound without the nitro group is a dull brown of little tinctorial value.

Nitrobenzidine

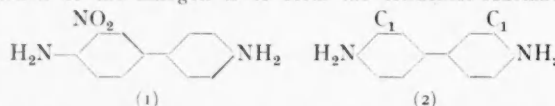
Contrary to the older opinions there is no need to acetylate benzidine prior to successful mono-nitration since the presence of a large excess of sulphuric acid will, as in the parallel case of benzaldehyde, act as a protection for the amino group. The apparatus for benzidine nitration may be similar to that used for the nitration of benzene, save that the cooling system need not be so elaborate, since the amount of heat generated is considerably less than in the nitration of benzene. The sulphuric acid (98 per cent. 3 cwt.) is piped into the nitrator and moist benzidine sulphate paste ($\frac{1}{4}$ cwt.—calculated on 100 per cent. dry benzidine sulphate) is added. The benzidine sulphate is added in the form of a paste since solution in the acid takes place so much more readily, presumably

owing to the heat of reaction between the residual water in the paste and the concentrated acid in the nitrator. The mixture is stirred until all the sulphate has dissolved and steam is run through the heating coil until the temperature reaches 55-60° C. This is necessary to ensure the solution of all the sulphate.

When solution is complete the temperature is lowered to 12-15° C. by means of cooling coils, and the nitration mixture is slowly run. The original patent (E.P. 13,475/1892) suggests the use of potassium nitrate for this operation, but nitric acid is usually much more convenient. The nitric acid (10 lb. sp. gr. 1.4) is diluted with four or five times its weight of sulphuric acid and run in so that the temperature does not rise above 20° C. The stirring is continued for five hours after the addition of the acid. The solution is then run into water (60 gallons) and allowed to stand for twenty-four hours. The sulphate of 2-nitrobenzidine (1) separate in the form of orange red needles, which carry with them half a molecular proportion of water of crystallisation. They are, unlike the sulphate of the un-nitrated base, moderately soluble in water, and are commercially used in this form.

Halogenated Benzidine Derivatives

The preparation of dichlorobenzidine is a more complex process than the manufacture of the mono-nitro derivative, since in the presence of the unprotected amino group the action of the halogen is to form the tetrachlor-substance,



which has no value commercially. This is prevented by the acetylation of the amino groups prior to halogenation. The acetylation is performed by boiling benzidine with glacial

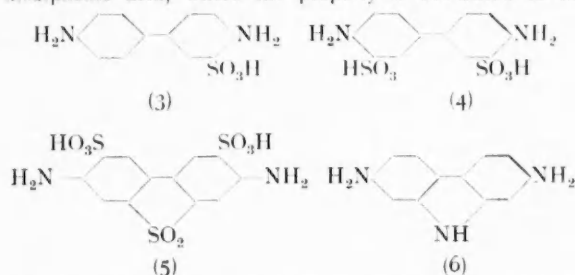
acetic acid in a vessel fitted with a reflux condenser through which water is only allowed to trickle slowly so that the water leaving the condenser has a temperature of about 80° C. The solid acetyl derivative without purification is dissolved in sulphuric acid (90 per cent. acid; 1½ cwt. for 56 lb. diacetylbenzidine), and it may be noted that the crude diacetyl benzidine should be finely milled before solution in the acid. The solution in acid is poured on to crushed ice in order to obtain a very fine suspension of the diacetylbenzidine particles. This suspension is maintained at 0° C. and sufficient clear hypochlorite of chlorine is run in to bring about the dichlorination of the diacetyl derivative, which towards the end of the run separates as a yellow granular crystalline material. Any excess of the chlorinating agent should be avoided since the process tends then to go on to the formation of higher derivatives.

It is interesting to note that at the commencement of this chlorinating operation the deep green colour so characteristic of reactions involving benzidine derivatives is formed, and disappears later, when the chlorination is more complete. Filtration is difficult, but by warming to 40° C. before the magma goes to the presses the separation can be readily accomplished. The product is the salt of dichlorodiacetylbenzidine and is converted to the corresponding compound containing the free amino groups by heating under reflux with concentrated hydrochloric acid. No definite time can be quoted for this operation since the rate of hydrolysis depends to a large extent on the particle size of the diacetyl derivative; with acid containing 20 per cent. of hydrogen chloride about three hours is necessary. The end point can be ascertained by drawing off a sample from time to time and diluted with water. When hydrolysis is complete the sample will be completely soluble in the diluted hydrochloric acid so formed. The final step differs according to whether the base or the hydrochloride is required; in the latter case the reaction liquor may be diluted with water until just clear and the hydrochloride salted out with sodium chloride in the usual way; in the event of the base being required, it may be set free by the slow addition of alkali which precipitated it as a fine white powder.

The filtration of the base is facilitated if the precipitation takes place from a moderately warm solution, which is allowed to stand for some hours after the base has come down. The base prepared in this way is very minutely crystalline and may, if desired, be purified by a further crystallisation when it is obtained in needles m.p. 133° C.

Benzidine Sulphonic Derivatives

There are only three sulphonic derivatives of benzidine which find any use in the preparation of dyestuffs on a commercial scale. They are valued for the fact that the azo-dyes from them give colours which are specially fast to milling, in comparison with those from the non-sulphonated derivatives. This is especially true of the benzidine sulphone-disulphonic acid, where the property is attributed to the



formation of the small secondary ring between the two *meta*-substituents. It has been pointed out that a similar ring exists in diaminocarbazole, a compound which forms strong substantive dyes in contradistinction to those from the corresponding nucleus in which the small subsidiary ring is replaced by one, or two, amino groups.

The heating of benzidine sulphate with strong sulphuric acid to temperature approaching 200° C. leads to the formation of the disulphonic acid. The almost dry benzidine sulphate is mixed with twice its weight of sulphuric acid (98 per cent.) and heated to a temperature 200-220° C. for 1½-2 days. The cooled reaction mixture is run into water and

then made faintly alkaline, when the benzidine-3:3'-disulphonic acid (4) goes into solution leaving the sodium salt of the monosulphonic acid as an insoluble residue from which the clear liquid is decanted. The final traces of the monosulphonic acid are removed by the addition of a little acetic acid, and after a second filtration the disulphonic acid is precipitated by the addition of an excess of mineral acid. About 85 per cent. of the benzidine disulphonic acid is obtained. If the benzidine-3-monosulphonic acid (3) is the object of the process, care must be taken not to use excess of acid, or a temperature above 180° C. The moist benzidine sulphate paste from the presses is mixed with dilute sulphuric acid into a thick cream. The amount of acid (calculated as 100 per cent. H_2SO_4 on the basis of 100 per cent. benzidine sulphate) is half the weight of the sulphate. The paste is evaporated to dryness, and spread on trays in a baking oven at 170-175° C. at which temperature it is maintained for one day. The product of this operation is a black porous mass which must be milled before further treatment can be carried out. The powder obtained by such milling is extracted with dilute alkali and filtered. The filtrate is made very faintly acid with lactic or acetic acids when the benzidine-3-monosulphonic acid is precipitated as the free acid.

Formation of the Sulphone Group

The use of oleum in the sulphonation leads to the formation of a sulphone group between the two aromatic nuclei. The *modus operandi* appears to involve the migration of the sulphonic groups from the "3" to the "2" position followed by the loss of one molecule of acid and the formation of the ring containing $-\text{SO}_2-$. In actual practice the sulphone may be obtained by warming benzidine sulphate with 20 per cent. oleum. The progress of the reaction is easily followed by the withdrawal of a small sample from time to time, which is diluted with water. When the reaction is complete the suspension so obtained fails to give the usual tests for benzidine. At this stage the solution is cooled, poured out into crushed ice and allowed to stand. The sulphates of benzidine and benzidine sulphone separate together, and that of the latter base is finally obtained pure by conversion to the base and extraction of the mixed bases with alcohol in which the sulphone is almost insoluble. It is very seldom that the separation of the simple sulphone is ever undertaken since it is almost invariably used in the form of its disulphonic acid (5).

The preparation of the benzidinesulphone-3:3'-disulphonic acid (5) is carried out by the simple process of warming benzidine sulphate (which in this case must be dry) with four times its weight of oleum containing 40 per cent. of SO_3 . The progress of the reaction is followed in the usual way and the temperature is kept at 100° C. until no more free benzidine sulphate is observable; the temperature is then raised to 155° C. and maintained there until sulphonation is complete, as judged by a test sample, which at the end of the reaction should give no precipitate on solution in water containing caustic soda. When this stage is reached the whole is poured out on to ice, a mixture of three substances separating, the sulphone and its mono and disulphonic acids, the latter preponderating. The precipitated mixture is taken up in alkali and the addition of lactic or acetic acid used to precipitate any monosulphonic derivative. After filtration the disulphonic acid is separated by the addition of a slight excess of mineral acid.

Scottish Dyestuffs Market

At the time of writing a more satisfactory report can be given than has been possible for some months. Business in the tweed trade is still improving. There is, of course, plenty of room for further betterment but an increasing number of operatives are being placed on full time. Orders for the next spring season are now coming in in a fairly satisfactory way. Meanwhile patterns are now being shown for the following winter season but it is a little early yet for orders to be expected. In the hosiers and knitted departments improvement is also to be noticed. Dyers are more fully occupied and a demand for dyestuffs is on the increase. At present there seems every prospect of the general improvement continuing.

Dyes and their Application: Recent Technical Progress

By L. J. Hooley

IN THE CHEMICAL AGE, *Dyestuffs Monthly Supplement*, July, 1931, some of the recent developments in the dyeing and processing of cellulose acetate were described. The number of patents dealing with this subject is large, and only a fraction of the more important ones were dealt with, these being the ones relating principally to dyes and to methods of dyeing. In the following notes other aspects of the dyeing and printing will be considered.

Mordanting and Weighing

In B.P. 336,621 (H. Dreyfus) a cellulose derivative is incorporated with a substance such as a metal salt by carrying out the spinning in acetone, in which a soluble metal salt has been dissolved and then treating with a precipitating agent, which may be done immediately after the spinning or in a coagulating bath or even at a later stage. Much handling and manipulation of material, and consequent risk of damage in weighing and mordanting processes, is avoided in this process. The material which has been mechanically impregnated with one of the reagents is not given a washing treatment, but is submitted to the action of the second reagent by mechanical impregnation, for example, in a padding mangle. For instance, after impregnating with a metallic compound the material may be "batched" and left to stand when the absorption of the metal compound takes place without any further manipulation; further by suitably adjusting the padding mangle the amount of the liquor which the goods retain can be controlled. The goods are then without washing subjected to a second impregnation with a precipitating agent. In an example, cellulose acetate, previously scoured and dried is treated in a padding mangle with a solution containing stannic chloride and ammonium thiocyanate, at 30° C. After batching on a roller the goods are allowed to stand for five hours at 30° C., with occasional turning, during which time most of the liquor is absorbed by the fibres. It is then passed, without washing, through a pad mangle containing a 30 per cent. solution of trisodium phosphate. After padding and washing, the goods show an increase in weight of 15 to 20 per cent. and have a full handle. (British Celanese, Ltd., and G. H. Ellis, B.P. 329,659.)

Previous impregnation with basic aluminium acetate or other basic metallic acetate, before printing, preferably with intermediate drying, leads to much sharper outlines in printing. An organic acid may be incorporated into the fabric along with the acetate with advantage. (B.P. 299,849, British Celanese, Ltd.)

Hydrolysis

Chlorazol Fast Helio BK may be used to detect saponification in cellulose acetate. It gives a pale blue on viscose but only a faint pink on cellulose acetate, so that on the partially saponified material, it gives an intermediate shade which serves as means of recognition. (A. J. Hall, *Rayon Record*, 1930, 801.) By hydrolysing cellulose acetate with alkali paste and using an alcohol such as glycol but not methyl or ethyl alcohol an effective result can be obtained with very much less than the quantity of caustic soda theoretically necessary, for example, sufficient alkali for 3 per cent. of the ester will bring about almost complete hydrolysis. (British Celanese Ltd., B.P. 313,404.)

Another patent by the same applicants dealing with hydrolysis is B.P. 309,377, where this is carried out in the presence of a soap and a salt of a strong base and a weak acid, the hydrolysing agent being added altogether at the beginning of the process as distinct from gradually as in an earlier application. A third patent by the same applicants is B.P. 313,971, where the hydrolysing agent is aqueous vapours of ammonia at 25 to 30° C. Although this treatment gives increased tensile strength and greater power of absorbing moisture it does not have the usual effect of hydrolysis or dyeing properties.

Pattern Effects

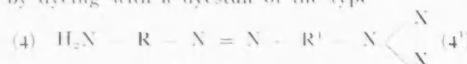
A fabric which has been delustrated with wet steam can be printed with a paste containing cellulose nitrate or other thickening agent insoluble in water together with a material

such as zinc oxide or barium sulphate as well as a dye and a plasticiser if desired together with ethyl alcohol or ether. The products are finally relustrated for example with acetic acid, the total result being a pattern effect of non-lustred portions on lustred portions. (British Celanese, Ltd., B.P. 314,396.) Another method of obtaining pattern effects is to first print the substance which will preserve the lustre and then give a delustrating treatment. As an example, goods may be printed with a solvent or galling agent and then after dry steamed with dry steam under pressure, and then boiled in soap when the printed portions resist the delustring. (*idem*, B.P. 335,583.)

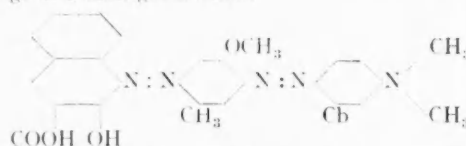
A device for lacol saponification consists in embossing the fabric and then printing with a roller which comes into contact with the embossed portion only. Such a treated material may be then dyed, for instance, with a substance which has a selective action towards either the treated or untreated portion. (British Celanese, Ltd., and R. S. Felgate, B.P. 323,210.) Matt effects can be made on cellulose acetate by printing with urea and a thickening agent and then drying, steaming, washing and drying. (I.G.F.A.G., B.P. 309,194.)

Dyeing, Printing and Discharging

Dark shades, such as navy-blue, black and dark green which can be discharged white can be obtained on cellulose acetate by dyeing with a dyestuff of the type



where R and R' are aryl residues as specified in the patent and X is an aliphatic residue, such as methyl, alkyl or hydroxyalkyl and then diazotising and coupling with β -oxynaphthoic acid. An example is the product shown below which gives a dark green shade.



It can be discharged pure white by the mono-zinc salt of formaldehyde sulphylic acid. 15,951/30, I.G.F.A.G.) Zinc salts of formaldehyde sulphylic acid are also recommended for discharging cellulose acetate in G.P. 461,753 (I.G.F.A.G.) In B.P. 334,543 the discharging process of B.P. 312,655, is modified by substituting chromates for chlorates (British Celanese, Ltd.).

Pattern effects in printing where the colouring is effected by processes of the Aniline Black type, using oxidisable amines, can be carried out by first printing with an oxidation inhibiting substance, then applying the oxidising agent and then the colouring substance. (B.P. 324,683, British Celanese, Ltd., and G. H. Ellis.)

P. E. King and A. R. Sahasranam, discuss the absorption of tannic acid by cellulose acetate. This was a point on which there had been some dispute and the authors find that contrary to the generally accepted view, cellulose acetate silk does absorb tannin to a considerable extent, even as much as half that absorbed in the case of viscose. The behaviour of the silk which has absorbed the tannic acid with basic dyes is somewhat peculiar, for instance, although it combines with Rhodamine B and Rhodamine 6G it does not do so with Malachite Green, the tannic acid having no effect on the depth of dyeing with this last colour. (*J. Soc. Dyers and Col.*, 1930, 118.) In B.P. 308,780 (I.G.F.A.G.) dyeing with chlorinated cellulose esters is described, the dyeing being effected with basic dyes. The chlorination considerably enhances the affinity.

Cellulose acetate pastes containing colouring materials can be used as printing pastes with textile fabrics. The method is claimed to have the advantage of requiring no after treatment, of being applicable to all materials and of giving very fair fastness to water, washing and rubbing. (Kemmerich, *Textilber.*, 1929, 950.) In B.P. 333,236 (S.D.I., J. S. Wilson,

E. G. Beckett and J. Thomas) the production of dry acetyl-silk dyestuffs of the anthraquinone series is described; as an example an aminoanthraquinone colour in the form of a finely dispersed paste of 10 per cent. strength may be mixed with a neutral soap and dried in a vacuum steam jacketed iron pan provided with a stirrer.

Puckering Effects

Crease marks which are liable to appear when cellulose acetate is wet-processed in rope form, can be avoided by first thoroughly wetting the material in open width for several hours preferably in the presence of a wetting agent. (B.P. 338,190, British Celanese Ltd. and G. H. Ellis).

Crinkling and puckering effects can be obtained by spraying materials which have been woven or knitted with a dilute solution of nitric acid, leaving to stand for a few minutes and then washing off. Shrinkage occurs where the acetate silk has come in contact with the fibre and this is responsible for the resultant puckering. (B.P. 335,138, British Celanese, Ltd.) By weaving from yarns some of which have been partially shrunk by a treatment with nitric acid similar to that just mentioned, and then repeating the treatment after weaving, crepe materials can be obtained. (B.P. 335,180, British Celanese, Ltd., W. A. Dickie and R. W. Moncrieff.) White or coloured matt surfaces can be obtained either on ethers or esters of cellulose by soaking with a mixture of urea and water and then drying and steaming. (I.G.F.A.-G, 345,673.)

Determination of Dyestuffs

New Methods Proposed

NEW methods for the determination of dyestuffs was the subject of a paper by S. R. Trotman and T. B. Frearson, at a meeting of the Nottingham Section of the Society of Dyers and Colourists, held at University College, Nottingham, October 14. The methods discussed were applicable to acid, direct and basic dyestuffs, and were claimed to be easy to manipulate, giving reliable results.

All direct dyestuffs give insoluble compounds with alkaloids such as quinine, cinchonine and strychnine. In the case of cinchonine these have the composition represented by the formula $(\text{RSO}_3\text{H})_2\text{B}$, where B represents the alkaloid and RSO_3H the dyestuff. These compounds can be filtered off on a Gooch crucible, dried and weighed. When the molecular weight of the dyestuff is known and the number of salt-forming groups contained in the molecule a factor for conversion of the precipitate into the dyestuff can be calculated. Neutral dyeing acid dyestuffs behave as a rule in the same manner as direct dyestuffs, but ordinary acid dyestuffs are either not precipitated by alkaloids or, if they are, the precipitate is insoluble in dilute acetic acid. Since the alkaloidal compound of direct dyestuffs is insoluble in dilute acetic acid the separation of acid and direct dyestuffs or of ordinary and neutral dyeing acid dyestuffs may be effected in many cases. For basic dyestuffs it was proposed to take advantage of the fact that they form insoluble compounds with silico-tungstic acid, $\text{S}_2\text{O}_5 \cdot 12\text{WO}_3 \cdot 2\text{H}_2\text{O}$. If the precipitate is filtered off on a Gooch crucible, washed, dried and incinerated a residue of $\text{S}_2\text{O}_5 \cdot 12\text{WO}_3$ is obtained from the weight of which the quality of dyestuff present in the solution can be calculated. Examples of analyses made by these methods were given and reference made to other applications of the methods, such as the determination of sulphates and chlorides in the presence of complex sulphonic acids.

Effect of pH on Photochemical Decomposition of Silk

THE effect of pH on the photochemical decomposition of silk is being determined at the United States Bureau of Standards by a research associate from the American Association of Textile Chemists and Colorists. Although silk is one of the strongest fibres, its susceptibility to attack by light has been the source of much trouble, particularly when used for window curtains or other articles that are exposed for a long time. The rate of weakening of silk which has been treated with solutions of different hydrogen ion concentrations was determined with the aid of an accelerated light ageing test. Silk treated at a pH of about 10 exhibited maximum stability. Above pH 11 and below pH 3, the stability decreases rapidly. In the neutral region pH 6 to pH 8, silk is less resistant to

light than when more acid or alkaline. This suggests that the silk protein is less stable than its salts. Contrary to what might be expected from the fact that alkalies hydrolyse proteins more rapidly than do acids, silk treated with tenth normal sodium-hydroxide solution is more stable in light than either untreated silk or silk treated with tenth normal sulphuric acid solution. The acid-treated silk is least stable. These facts have an important bearing on practical silk finishing as well as on the nature of the action of light on silk.

Photosensitising Dyes

[To the Editor of THE CHEMICAL AGE.]

SIR,—In the article on Basic Intermediates for Dyestuffs, No. XLV, Quinoline and Related Compounds, THE CHEMICAL AGE, September 12, page 15, there are several points which call for attention. While no fundamental distinction is drawn between visible and near infra-red radiation when dealing with the photographic sensitising action of Neocyanine, some differentiation seems to have been used in the consideration of Dicyanine A, and it is misleading to state that "it is not a photosensitising dye."

Again, the dyes of the indocarbocyanine group, such as Astraphloxine FF, possess photosensitising properties, although a statement is made to the contrary. The photographic action of 1:1'-diethyl-3:3':3':3'-tetramethylindocarbocyanine iodide was described by Bloch and Hamer (*Phot. J.*, 1928, lii, 21) who showed that it gave a band of extra-sensitisation with a maximum at about 5800 Å. The lack of green sensitisation imparted by Kryptosyanine is by no means a "unique feature," since it is shared by all the other infra-red sensitisers of the cyanine series. Even with a strong red and green sensitiser such as Pinacyanol, there is a pronounced minimum between the blue and the green, and as the sensitising band moves in the direction of longer wavelength this minimum broadens, until with the infra-red sensitisers there is a wide gap between the regions of ordinary sensitivity and conferred sensitivity.

Lastly, it should be pointed out that Cyanine was first discovered by Greville Williams in 1856 (*Jahresbericht d. Ch.*, 1856, 532; 1857, 400; 1860, 361), and not by Hoogewerff and van Dorp in 1883. The last named authors, however, discovered the isocyanines, (*Rec. Trav. Ch.*, 1883, ii, 41, 317; 1884, iii, 337) independently of Spalteholz (*Ber.*, 1883, 16, 1847).—Yours etc.,

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New Colours for Shoe Leathers

NINE high-style colours and five classic staple shades for women's shoes will appear on the 1932 Spring Shoe and Leather Card to be issued shortly by the Textile Colour Card Association. These official colours were selected by the joint colour committee of the Tanners' Council of America, the National Boot and Shoe Manufacturers' Association and the National Shoe Retailers' Association, in co-operation with the Textile Colour Card Association. The new "high style" colours are fawn brown, sunrust and platine; the latter is a clear light grey. In addition, there is a special group of six shades termed "sun colours," which includes shell (a very new development in shoe fashions, so-called because it reflects the soft blush tint of the interior of a shell); Biscay blue (a bright medium blue, slightly purplish in cast); tropic yellow, Cabana green and orange glow.

Japanese Dyestuffs Trade

ACCORDING to the Japanese Coal Tar Chemical Industry Association, the 1930 Japanese output of dyestuffs was as follows: Basic colours 257,633 kilos; direct colours 609,363; acid colours, 239,627; acid mordants, 41,568; mordant colours, 20,783; sulphur colours, 6,472,947; vat colours, 94,452; dyestuffs soluble in oil, artificial indigo excluded, 43,993.

China's Aniline Dye Imports

IMPORTS of aniline dyes into China through Shanghai increased from a value of 1,928,000 customs gold units (about 2s.) in the first six months of 1930 to 3,110,000 C.G.U. during the corresponding period of 1931. Of the 1931 six months imports, Germany supplied a value equivalent to 2,064,652 customs gold units.

Dyestuffs from Fluorenone

Possibilities Investigated

Two Indian chemists, A. C. Sircar and K. C. Bhattacharyya, have recently investigated the possibility of preparing dyes from fluorenone by taking it as a chromophoric seat and packing other groups round it. Hitherto no dyestuff had been prepared from fluorenone except those described by Underwood and Kochmann (J. Amer. Chem. Soc., 1923, **45**, 3075). Their researches are recorded in the September-October issue of the *Journal of the Indian Chemical Society*.

The $\text{N}=\text{CH}$ group, like the azo-linking $\text{N}=\text{N}$ is known to enhance tinctorial properties (Green and Sen, J. Chem. Soc., 1910, **97**, 2242; Morgan and Reeves, J. Chem. Soc., 1922, **121**, 1; Sircar and Sen-Gupto, J. Indian Chem. Soc., 1924, **1**, 321) and successful attempts have been made of creating the azo-methine groups in the fluorenone molecule by condensing various aromatic aldehydes, e.g., benzaldehyde, salicylaldehyde, m-hydroxybenzaldehyde, p-hydroxybenzaldehyde, m-nitrobenzaldehyde, o-nitrobenzaldehyde, p-nitrobenzaldehyde, p-acetylaminobenzaldehyde, p-dimethylaminobenzaldehyde, resorcydaldehyde and vanillin with 2-aminofluorenone, the object being to investigate the effect of azo-methine groups on the colour of the compound.

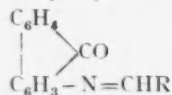
Azo Derivatives

Fluorenonephenylazo-methines are easily obtained and many of them are fairly deep coloured and form well-defined crystals, but are not satisfactory so far as their dyeing properties are concerned. They possess no affinity for cotton and are decomposed into the original components when boiled for some time with water in presence of an acid. Wool can however be dyed with them from one per cent. acetic acid bath between 80 and 85°. But though the bath is exhausted, even in that case the shades obtained on wool are neither very bright nor very deep. In their properties therefore the fluorenonephenylazo-methines closely resemble the phenanthraquinonephenylazo-methines described by Sircar and Sen-Gupta.

A number of azo-derivatives have also been prepared by coupling 2-diazo-fluorenone chloride with various phenols and amines e.g. β -naphthol, dimethylaniline, phenol, salicylic acid, 2-hydroxy-3-naphthoic acid, naphthionic acid, 1-naphthol-4-sulphonic acid, diethylaniline, R-acid (β -naphthol-3:6-disulphonic acid) and G-acid (β -naphthol-6:8-disulphonic acid). The fluorenoneazo-derivatives are obtained mostly in well-defined crystalline form and majority of them are deep coloured and possess well-developed dyeing properties. They dye wool evenly from one per cent. sulphuric acid bath in shades ranging from orange yellow to deep scarlet and bluish-violet.

Preparation of Azo-methines

For the preparation of the azo-methines equimolecular proportions of 2-amino-fluorenone and the aldehyde are dissolved in the least quantity of alcohol, except where otherwise mentioned, and the solution heated on the water-bath until the separation of the solid is complete (the time required for the same has been noted in each case). The product is purified by crystallisation. The yield is generally almost theoretical. They are moderately soluble in alcohol, sparingly soluble in acetone or chloroform and insoluble in water and have the following general formula, where R = phenyl or substituted phenyl nucleus.



For the preparation of the fluorenoneazo-derivatives 2-amino-fluorenone is diazotised (Diels, Ber., 1901, **34**, 1764) and the 2-diazo-fluorenone chloride is coupled with the calculated quantity of the second component in the usual way.

Attempts to Prepare Vat Dyes

An attempt was also made to prepare vat dyes from 2-amino-fluorenone by condensing it with various dibasic acid chlorides, e.g., oxalylchloride, phthalylchloride, carbonyl chloride, thionyl chloride, malonic acid (in presence of phosphorus pentachloride), succinic acid (in presence of phosphorus pentachloride) and glutaric acid (in presence of

phosphorus pentachloride), but contrary to the expectation, the resulting fluorenoneylamides could not be made to yield soluble vats with hydrosulphite. They however dye wool, though slowly, from a one per cent. sulphuric acid bath. But the shades obtained, though even, are invariably very light and rather disappointing. Attempts to apply Ullmann's reaction to 2-bromofluorenone (Schmidt and Bauer, Ber., 1905, **38**, 764) with a view to prepare anilino-fluorenone were unsuccessful in the sense that simultaneously with the bromine atom the ketonic group was also attacked.

The condensation of the dibasic acid chlorides with 2-amino-fluorenone is easily affected by heating for about two hours a solution of two molecules of the aminoquinone with somewhat more than one molecule of the acid chloride in nitrobenzene. The condensation products are precipitated from the nitrobenzene solution on the addition of ether and are purified by repeatedly washing with ether and finally with water. They resist all attempts towards crystallisation, being insoluble in all organic solvents, and are reddish-brown or brownish-red amorphous bodies, not melting below 200°.

History of a Dyed Dress

Alleged Poisoning by Para-amino-phenol

A MASS of scientific evidence was taken on Friday, October 30, in the Civil Court at Liverpool Assizes, concerning the history of some marocain dyed in Switzerland. The case was that in which Miss Hilda Margaret Robinson, of Alexandra Court, Alexandra Drive, Sefton Park, originally obtained £707 damages against Miss Ellen Hartley, costumier, of Bold Street, Liverpool. George Oakenfold, dress manufacturer, of Paper Street, London, fourth party in the action, now asking for an indemnity against the fifth party, Heer and Co., of Thalwil, Switzerland, from whom he obtained the marocain to make the dress.

Professor Frederick Rowe, of Leeds, said he tested a piece of the dress and silk trimming and found no indication of para amino phenol.

Dr. Henry Cox, public analyst for Hampstead, said that in his whole experience he had never known a case of dermatitis to be due to the use, in a textile fabric, of para-amino phenol as a dye. It was not, in fact, a dye. In a piece of Miss Robinson's dress and the silk trimming he found in them no trace of para-amino-phenol. He had made a test by wetting pieces of the material and placing them on his arm for forty-eight hours. Except for a slight blue stain there was no result.

Evidence for the defence was also given by Dr. Schofield, the dye expert, of Manchester College of Technology.

George Schwyzzer, director of Stuckfarberei, Zurich, described the dyes he used, which came, among other sources, from the Society of Chemical Industry at Basle, and from "I.G." in Germany. Between 1922 and 1927 he dyed for Heer's over 100,000 yards with the dye, and he had used it in dyeing for other people to the extent of over 600,000 yards without a single complaint.

Dr. E. Anderwert, of Basle, manufacturers' representative, said their workmen had handled the dyestuffs and none had suffered any ill effects from them or their component parts. In about ten years they had made enough dyestuff to dye 500,000,000 yards of material, and they had had no complaints.

Mr. J. Whitehead, K.C. (for Heer's), told the jury that the evidence he had called related to experience for fur dermatitis because there was not a "solitary soul" who had had experience of dermatitis contracted through para-amino-phenol on these textiles, which it was not used to dye.

Colour Users and the Dyestuffs Act

In Favour of Continuance

THE Council of the Colour Users' Association on Friday, November 6, unanimously adopted the following resolution:

"That the Colour Users' Association consider that any tariff on dyewares—one of their important raw materials—would be prejudicial to their industries and whilst not receding from their declarations regarding the Dyestuffs (Import Regulation) Act, they agree that in present circumstances a continuation of that Act for a further twelve months would be preferable."

Dyestuffs Monthly Supplement

Published in the second issue of "The Chemical Age" each month

Communications relating to editorial matter for the *Dyestuffs Monthly Supplement* should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Advertisement matter, subscriptions, etc., should be sent to the Manager. The Supplement is devoted to the interests of both manufacturers and users of dyestuffs, and contributions will be welcomed.

The Dyestuffs Act

Since the November issue of the *Dyestuffs Monthly Supplement* appeared the fate of the Dyestuffs (Import Regulation) Act has been decided by its inclusion in the Expiring Laws Bill. A short debate took place in the House of Commons on November 23, when the House went into committee on this Bill. On that occasion Major Attlee, the Deputy Leader of the Opposition, moved an amendment that the Dyestuff Act be omitted from the list of Acts to be continued, but he did not press the amendment to a division. British dye producers, he asserted, were concentrating on a small number of dyes; hence a large amount of importation still took place. He produced no arguments against the renewal of the Act, nor could he give any valid argument in favour of throwing open our industry to unrestricted imports of foreign dyes at a time, moreover, when excess of imports is one of our national problems. Mr. Hore-Belisha, Parliamentary Secretary to the Board of Trade, however, brought forward figures to show that great progress has been made by the British dyestuff industry since the Act came into force. At this juncture, he said, we should not be safe in allowing the textile industry once again to become dependent on foreign countries. The number of dyes we could not produce in this country was diminishing every year.

World Colour Pact

British users of colours and textile merchants and manufacturers who deal in coloured yarns and fabrics are closely affected by any agreement concluded between British and Continental dyestuffs makers. Negotiations for such an agreement have been going on for some time, and it is now reported that an arrangement for the division of markets on a territorial basis has actually been reached. The Frankfort correspondent of the *New York Journal of Commerce*, in a special cable, announces that a world-wide agreement in the dyestuffs industry has been virtually perfected as a result of Britain's agreement to join the Continental dyes cartel, consisting of Germany, Switzerland, and France. The main terms of the agreement are that Germany and Switzerland have made certain concessions to Britain as regards the allocation of markets in India, and in return British makers have undertaken to curtail their competition in other markets. Under the new arrangement the German I.G. retains its market in Britain for high-priced specialities, but undertakes to leave business in other products to British producers. The agreement also provides for the interchange of information and new discoveries, as well as for the control of competition within the line of the present division of world markets among the chief companies. Previously Germany was allocated 70 per cent. of the total export trade in dyes, France 20 per cent, and Switzerland 10 per cent. With the inclusion of Britain, this arrangement will, of course, be revised.

The Egyptian Market for Dyes

The dyeing trade in Egypt is the subject of a very interesting chapter in a report on "Economic Conditions in Egypt," which has just been issued by the Department of Overseas Trade (H.M. Stationery Office, 4s. net). Coal tar dyes have almost completely replaced the natural

colours, which are now only used in very limited quantities for better class work. Synthetic indigo was first introduced into Egypt about 40 years ago by two German factories which regularly sent their chemists to Egypt, and every little village of the country was for years systematically canvassed. Thus the local industry was developed with petty dyeworks all over the country, which form a regular clientele for dyes and indigos of various strengths. Before the War, imports of coal tar dyes and synthetic indigo amounted to over £100,000 per annum. Since the War, however, the import of ready dyed cotton and silk materials has materially affected the primitive dyeing industry in Egypt. In 1926 the main German dyestuff producers amalgamated and established their own office in Cairo, under the management of their old agent. The following year the French producers joined the merger, and in 1928 a *de facto* sales control over Swiss dyestuff production was, states the report, attained by the combine. Italian producers have an agreement with a Paris firm, controlled by the German combine, and have abandoned the Egyptian market for indigo, but are still selling other dyes. In 1929, therefore, the German combine had full control of the dyestuff trade in Egypt and commenced increasing prices. The same year a British concern established a branch office in Cairo and commenced supplying dyes and indigo, and during 1930 the British range of dyes was introduced all over the country. Dyehouses of the primitive oriental type are to be found in almost every town and village in Egypt. In 1929 Parliament granted a considerable sum for the erection of model dyeworks in Cairo. A British expert is in charge and the work is controlled by the Department of Commerce and Industry.

The Spring Season Colour Card

The Spring Season Colour Card 1932 and Spring Season Wool Colour Card 1932, issued by the British Colour Council, contain 60 and 40 shades respectively, generally of a rather more striking character than in previous issues. Inquiries made from distributors, producers, and colour makers rather point to insufficient use being made of these carefully selected ranges of shades. At the same time it is noteworthy that several leading firms, both distributors of coloured textiles and producers of the same goods, are now issuing a card periodically in substitution of their own shade cards, and doubtless this lead will be followed by the smaller independent firms when they fully realise the considerable saving of both time and expense which this procedure means to them. The production of a pattern card, however modest, in most small concerns is an event of importance and entails an amount of time, thought and expense entirely out of proportion to its value to the producer in most cases. According to the current issue of the *Journal of the Textile Institute*, the textile producers of this country are slow to adopt any new idea until they are convinced that the innovation is likely to be of considerable benefit, and from the investigation carried out it appears that the points outlined above have not been fully considered by the trades concerned. If a suggestion might be made to the British Colour Council, co-option of feminine opinion before the production of the next issue would probably result in an improvement of the ranges and the cutting out of dead wood.

New Woollen Colours

New renditions of red, including subtle terra cotta and rust shades, as well as vivid scarlet and geranium types, figure prominently in the range of fifty colours which will appear on the Spring 1932 Woollen Card issued by the Textile Colour Card Association of the United States. Blues likewise occupy an important place, being reflected in clear navy and lighter than navy tones, soft slate versions and vibrant sports blues of slightly purplish cast. Beiges, as a high-fashion revival for Spring, are shown in various tonalities, the soft blush and vanilla tints being of major style interest. Champagne and sandy beiges represent the more staple types. A new bleached white is substituted for eggshell as a smart accent note with brown, blue or other basic costume colours. The green family is expressed in soft olive and slightly yellowish tones, bluish sea greens and virile sports hues.

A New Monolite Colour

Imperial Chemical Industries have just introduced a new fast yellow in paste and powder form as an addition to their range of Monolite colours. Monolite Fast Yellow LA 10GS is a pigment yellow dyestuff approximately equal in shade to Monolite Fast Yellow 10GS and possessing similar high grade fastness properties, except in respect of resistance to lime. It will be found eminently suitable for the production of high grade printing inks of all classes, oil paints, artists' colours, water paints and for the colouring of wallpapers, and surface papers where a brilliant fast to light greenish yellow shade is demanded. It is also well adapted for admixture with the Chrome Yellows to improve the printing and light fastness properties. The excellent fastness to light and caustic alkali renders this colour specially suitable for the manufacture of inks for soap wrapper printing and for exhibition carton-boards where the ink has to withstand the influence of light and very often the presence of alkali in the paper compound. It is insoluble in water and requires no precipitating agent. In use it is simply mixed or ground with the required substratum into the process medium. Its fastness properties are unaffected by acids, alkalies, oil, spirit, and the action of light. In contact with lime, the shade turns somewhat redder.

The Free Acid of Orange II.

Isolation of Metallic Salts

SOME years ago, in the course of a study of the effect of hydrogen ion concentration on the so-called adsorption of dyes by hydrous oxide gels, Marker and Gordon (*Ind. Eng. Chem.*, 1924, **16**, 1186) remarked certain phenomena which were interpreted at that time as indicative of compound formation. Some time later Weiser and Porter (*J. Phys. Chem.*, 1927, **31**, 1704) questioned these conclusions upon the basis of the improbability of forming an appreciably soluble salt from the much less soluble gel (hydrous alumina) and "a weak, slightly soluble acid" (free acid of Orange II) and described the product analysed by Marker and Gordon as a mixture of alumina gel and the "insoluble acid" in equivalent ratio. Although the remarks of Weiser and Porter involved certain obviously erroneous assumptions they appeared to cast doubt upon the validity of the conclusions presented in the original publication, if not upon the good faith of the original investigators. Accordingly, it seemed desirable to carefully re-investigate the whole matter by means of a procedure so detailed as to leave no room for ambiguity in the results obtained. The results of this re-investigation were presented before the Dye Division of the American Chemical Society at the Indianapolis Meeting this year, and have now been published in the *American Dyestuff Reporter*, October 12, the joint authors being N. E. Gordon, G. Seidel, B. W. Allan and C. M. Loane, all of the John Hopkins University.

Adventitious Colouration in Raw Wool

Three types of adventitious colouration are met with in raw wools, according to a recent article by A. M. Stewart and C. Rimmington (*Wool Record*, 39, 985). One type is due to foreign materials—e.g., coloured soils, dirt, etc.; a second is due to growth of pigment-producing bacteria or micro-organisms on the wool, these being usually greenish-blue or red; and the third is pigmented wool coloured by secretions or pigments produced by the animal. True "canary stain" is a bright canary colouration which persists after scouring. It is probably of bacterial origin, although this is not certain. It is often confused with the dull golden colour associated with wool showing the characteristic termed "dead yolk." The authors propose to term this "golden colouration." It is completely removed by scouring. It is sometimes evenly distributed along the staple and sometimes occurs in bands. The yolk appears opaque and has a cheesy consistency. Apart from questions of yield, it is not objectionable. It bears no relation to the amount of ether-soluble grease in the wool, but there is a definite relation between intensity of colouration and proportion of water-soluble suint. The pigment responsible for "golden colouration" has been isolated and named "Lanaurin." It is probably secreted in the sweat and may afford some protection to the fibre from the damaging effect of short light waves.

Dyestuff Licences for November

The following statement relating to applications for licence under the Dyestuffs (Import Regulation) Act, 1920, made during November has been furnished to the Board of Trade by the Dyestuffs Advisory Licensing Committee:—The total number of applications received during the month was 666, of which 518 were from merchants or importers. To these should be added 7 cases outstanding on the 31st October, making a total for the month of 673. These were dealt with as follows:—Granted, 608 (of which 595 were dealt with within 7 days of receipt); referred to British makers of similar products, 46 (of which 44 were dealt with within 7 days of receipt); outstanding on November 30, 19. Of the total of 673 applications received, 639, or 95 per cent., were dealt with within 7 days of receipt.

Aluminium and Iron Salts

The conclusion drawn is that the free acid of Orange II behaves as a fairly strong monovalent acid. In supposed adsorption studies in which Orange II is brought into contact with amphoteric gels in acid media it is necessary to bear in mind the probability of chemical interaction. The alumina salt of the dye acid was prepared under various conditions, but when the study was extended to the reaction of the dye and the dye acid with ferric salts and with hydrous ferric oxide gel, several complications were encountered.

Since it seemed desirable to avoid the mutually destructive action of the ferric iron and the dye radical it was decided to carry out as many as possible of these reactions in the cold, which precluded the possibility of obtaining a crystalline product. In effecting the interaction of the dye acid with hydrous ferric oxide, it was, however, impossible to proceed in the cold, for the product cannot be separated from the gel save by solution, filtration and precipitation on cooling. Ferric chloride equivalent to one part was dissolved in a small amount of water and precipitated with an excess of ammonium hydroxide. After boiling to remove the excess of ammonia, the precipitated hydrous ferric oxide was filtered out on a small Buchner funnel. Gel and paper were then added to one litre of boiling solution containing double the amount of the dye acid calculated as equivalent to the F^{II} taken. The mixture was thoroughly stirred and then filtered through a large Buchner filter. The filtrate was quickly cooled and the fine crystals which separated constituted the product.

The Protection of Wool in Alkaline Liquors

By Dr. Kehren

The following article is abstracted from a recent issue of "Melliand Textilberichte" (Heidelberg). The author is associated with the Dyeing Department of the Prussian Höhere Fachschule für Textilindustrie, M.-Gladbach.

It is well enough known that wool fibre is very sensitive to the action of alkalis, indeed a strong solution of alkali will completely dissolve the substance of the wool, while it is more or less seriously injured by alkali according to the concentration of the solution, its temperature, and the length of time it is exposed to the action of the alkali. Even quite weak solutions may make the fibre hard or strawy if they are not properly applied, while a strong solution or a weak solution at a raised temperature will not only produce a harsh feel, but will also injure the fibre, leading to loss of strength or even making it brittle. Since even a slight harshness of the fibre is accompanied by a loss of quality of the finished goods, special precautions are advisable in manufacturing operations wherever wool has to be brought into contact with alkalis in the finishing process.

Metasal K is a light brown, viscous solution which consists of a soap that is not affected by lime and decomposition products of albumen. A combination of this type must be called very fortunate, because the soap contained in it has a detergent effect, while the albuminous bodies protect the wool fibre against all injury by alkalis and the moist heat of raised temperatures. Metasal K is quite simple in its application, because it can be added at once to the scouring liquors owing to its being readily soluble in water and alkalis.

Scouring Dirty and Greasy Wool

The quality of the water is an important factor in every washing process and the results obtained depend upon it, so that it is essential to use water that is as soft and free from iron as possible especially for scouring wool, which is so sensitive. Particular attention must also be paid to the composition of the scouring liquor when washing wool, if the valuable properties of the fibre are to be preserved. Care must be taken to avoid not only too high a concentration of the alkali, but especially also too high a temperature of the scouring liquor. On the other hand, however, it is advisable to raise the temperature of the liquor above the melting point of the wool fat, which is thus better emulsified and the scouring effect is improved. For this reason, mills generally carry out the scouring at a temperature of from 40 to 45°C (104–113°F), whereby certain precautions must be adopted in order to prevent injury to the fibre.

Certain classes of wool require either more alkali or a higher temperature in order to remove the grease to a certain extent, so that Hungarian or Cape wool, for example, needs

more soap and soda if it has to be washed at a low temperature. This results mostly in a yellowing of the fibre which is often accompanied by shrinkage, whereby the wool loses some of its whiteness and fineness. Thus if the strength, softness and spinning qualities of wool fibre are not to suffer from the alkali of the scouring liquor, an efficient fibre protecting agent must be added in all cases, or at least wherever the alkali must be highly concentrated or the scouring has to be carried out at a temperature above 40°C.

Wool that has been washed with the addition of Metasal K appears more open, finer and whiter than when merely handled through soap and soda. It has proved specially valuable in mills that operate small washing machines or have only a scouring and rinsing bowl at disposal. Owing to the protective action of the decomposition products of albumen the temperature of the scouring liquor can be raised to as much as 60°C (140°F) without any fear of injuring the wool. It is thereby possible to emulsify the yolk to such an extent after a short steep of about a quarter of an hour that the wool appears perfectly scoured after having been well squeezed out and rinsed.

Washing Woollen Yarn

Difficulties are very liable to occur in the washing of woollen yarn if it exhibits the dreaded property of tending to felt, such as is often met with in fine zephyr yarns, or if the yarn is very dirty. Felting is caused not only by the mechanical handling during the washing process, but is also promoted by the injurious effect of alkali upon the sensitive fibre.

The troubles that make their appearance in dyeing scoured woollen yarn are often due to the grease left behind in the fibres, which mostly originates in the raw wool, but may also consist of lubricating oils that are either not saponifiable or only with difficulty, or may be caused by insoluble lime soaps due to the use of hard water. These impurities adhere very firmly to the fibres and the wool must be energetically washed in order to remove them, an operation which is only in a position to withstand if a suitable fibre protecting agent has been added to the washing liquor, presuming that the alkali content has been raised and the temperature is kept rather high in order to secure good results. The addition of Metasal K in such a case also has proved very effective, not only because it permits of the use of strong alkaline liquors, but also because the felting caused by injury to the fibre is reduced to a marked extent.



FIG. 1. NORMAL WOOL FIBRES AS SEEN UNDER THE MICROSCOPE.

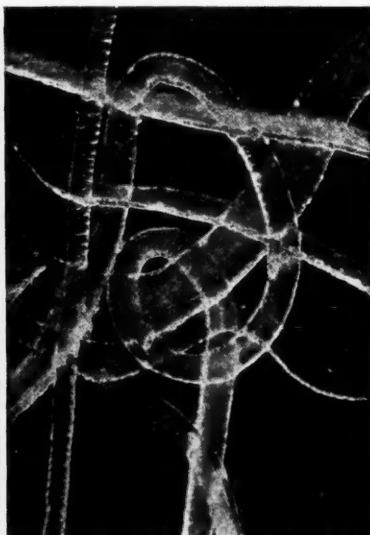


FIG. 2. WOOL FIBRES INJURED BY TREATMENT WITH ALKALI.

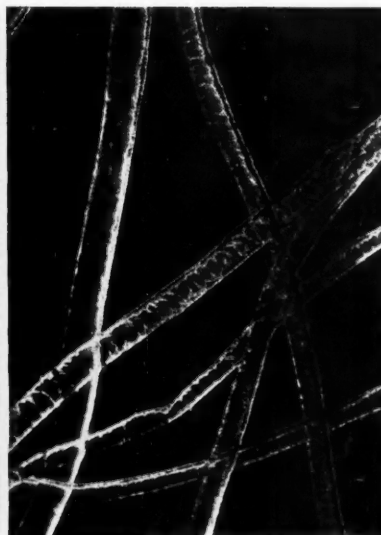


FIG. 3. WOOL FIBRES TREATED WITH ALKALI AND METASAL K.

Washing and Milling of Piece Goods

After the goods have left the loom they must often be thoroughly washing in order to remove lubricating oils and other impurities. If the fibre contains sufficient quantities of a saponifiable lubricating oil (olein), the washing process merely requires the addition of a corresponding amount of alkali to the washing liquor (such as soda or ammonia), otherwise soap must also be added. The more expensive washing with ammonia is preferred for goods of better quality and it has the advantage that the wool fibre is treated very gently, but as a general rule cheaper goods are washed with soda which, as is well known, easily injures the fibre. That is to say, if the washing liquor contains too much soda, or if it is dilute enough but is too warm, the alkali will injure the wool fibre and the damage will make itself evident in many different ways, for instance, the wool loses its soft handle and the piece goods are harsh and brittle, white goods turn yellow, the white of white mixtures is not clear, and so on. In order to avoid all injury by alkali, it is recommended to add Metasal K to the washing liquor, whereby the wool fibre is protected to a very great extent.

The aim of the milling process is to felt together the fibres that lie loosely side by side by mechanical means without injuring them, the felting being more or less pronounced as demanded by the quality of the goods. Although a certain degree of heat assists the milling process, care must be taken to prevent the goods from becoming too hot, which would make them shrink unnaturally. In such a case also Metasal K has a beneficial action, because it not only eliminates the injurious shrinkage, but at the same time also promotes the milling process.

Crabbing and Boiling Worsted Goods

It very often occurs during the crabbing and boiling of worsted piece goods that the pure water used for the purpose takes up residues of alkali left in the cloth from the preliminary scour or from the size, which cannot always be avoided in spite of the most careful rinsing. The consequence is that the wool is attacked by the bath, which is now alkaline, and the injury is more pronounced the more cloth is being handled. The results of the alkaline attack make themselves most evident when the cloth is being dyed later on, for example, the cloth last boiled may dye a deeper shade than the goods first treated, because wool is inclined to dye darker, the more it has been injured. This drawback can easily be prevented by making an appropriate addition of Metasal K to the water, since this fibre protecting agent hinders injury to wool fibre even in a boiling solution of alkali.

The finishing of woollen mixture fabrics containing cotton, pure silk, or artificial silk also demands treatment in a fairly strong alkaline bath, perhaps even at the boil, which can naturally only be done if a reliable fibre protecting agent is added to the liquor. If mixture fabrics of wool and silk have to be de-gummed it is recommended to replace half of the soap required by Metasal K, whereby the wool is prevented from shrinking too much and turning yellow.

When mixture fabrics of wool and cotton (union felts) have to be dyed pale shades, they must first be bleached. A mild bleach which will not attack the wool usually takes a very long time and has further the disadvantage that the cottonseed husks cannot be entirely removed. A strong bleach, on the other hand, attacks the wool fibre too strongly. A perfect bleach, however, can be produced within three hours at a temperature of from 55 to 60°C with a bleaching liquor containing 10 grams perborate and 5 grams Metasal K per litre. The liquor is thereby only half exhausted and can be further used after having been brought up to strength. Exact figures indicating the exact quantities of Metasal K in each case have not been given in the foregoing general sections 1 to 5, because they must be adapted to the prevailing operating conditions, but any mill man should be able to select the appropriate working methods by the aid of the special recipes contained in the various pamphlets issued by the manufacturers.

Proof of Protective Action

Although mill practice has long ago proved the far-reaching protective action of Metasal K, the exact scientific proof has also been brought forward by the labours of the Deutsches Forschungsinstitut für Textilstoffe in Karlsruhe (German Research Institute for Textiles).

These tests were carried out on material described as "pure white tops A" in hot alkaline baths with and without the addition of Metasal K. After having been rinsed and dried the material was tested for any injury to the fibres and comparative strength tests were carried out. For test No. 1 10 grams top was treated in a solution of 1 gram soda ash per litre for half an hour at 95°C; test No. 2 in a solution of 1 gram soda ash and 5 grains Metasal K per litre for half an hour at 95°C; test No. 3 in a solution of 30 grams soda ash and 20 grams soap per litre for 2 hours at 40°C; test No. 4 in a solution of 30 grams soda ash, 10 grams soap and 10 grams Metasal K per litre for 2 hours at 40°C.

A Pauly's familiar diazo reaction* served as microchemical proof of the presence of damage to the fibre caused by the action of alkali. This reaction is positive only at those parts of the wool fibre where the cementing substance of the epidermis cells has been injured and loosened mechanically (cracks, cut ends, and the like) or chemically, whereby the tyrosin of the fibre thus exposed is tinted by the soda-alkaline solution of diazo-benzol-sulphonic acid. The colouration produced by that reagent ranges from orange-yellow through yellowish red to deep blood-red, according to the extent of the damage caused by alkali or by acid, while intact wool fibre remains colourless even after prolonged exposure to the reagent for half an hour.

The test was carried out by laying the fibres that had been taken from the hanks of wool into the freshly prepared reagent for ten minutes. They were then examined under the microscope and classified by the following method according to the extent of the colouration:—

- Group 1. Fibres remain quite uncoloured (unless for an unavoidable red tinge at any cut ends that may happen to be present).
- Group 2. Fibres with red coloured ends.
- Group 3. Fibres with red coloured ends and a few red patches lengthways.
- Group 4. Fibres with from one-eighth to one-quarter of their length coloured red.
- Group 5. Fibres with from one-fourth to one-half of their length coloured red.
- Group 6. Fibres with more than half of their length coloured red.

Photomicrographs

After the various alkaline washings, 300 fibres were taken from each hank, treated with the diazo reagent, examined under the microscope, and allotted to one of the six groups, according to the extent of the injury, the percentage of fibres belonging to Groups 1—3 and Groups 4—6 being calculated as follows:—

	Groups	
	1—3	4—6
Untreated wool	99 %	1 %
Washing test No. 1	86.6 %	14.4 %
Washing test No. 2	88.7 %	11.3 %
Washing test No. 3	60.8 %	39.2 %
Washing test No. 4	71.4 %	28.6 %

From these figures it is perfectly evident that wool that has been treated in a washing liquor containing Metasal is much less injured than wool treated in an alkaline liquor of the same concentration under exactly the same conditions, but without the addition of a protective agent.

The excellent protective action of Metasal K can be demonstrated by means of photomicrographs. Fig. 1 shows intact wool fibres with the characteristic scales lying over one another like the tiles of a roof. Whenever the fibre is injured by alkali, important structural changes set in which vary in extent accordingly to the damage done and become evident externally in the way described in the preceding lines. In Fig. 2 it can plainly be seen that wool injured by alkali has lost its scales, the fibres are swollen and have assumed a glassy appearance. In contrast, Fig. 3 shows wool fibres that have retained their normal appearance and their other valuable properties in spite of severe treatment with alkali, after the addition of Metasal K.

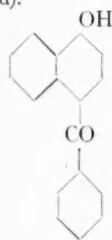
*Two grams of sulphanilic acid are suspended in 3 c.c. of water and 2 c.c. of concentrated nitric acid and then carefully diazotized with 1 gram sodium nitrate dissolved in 2 c.c. of water. The diazo-benzol-sulphonic acid thereby formed is lightly washed, collected on a filter, and finally dissolved by pouring over it a 10 per cent. solution of soda. The reagent prepared in this way decomposes readily so that the solution must always be freshly prepared for use as required.

Developed Dyes: Recent Technical Progress

By L. J. Hooley

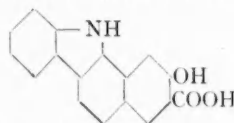
WHILE the azo colours form one of the two most important branches of dyestuff chemistry at the present time, the developed or ice colours is the section to which most attention is being paid at the moment, and in spite of the considerable amount of work which has been carried out since the war, new results of considerable interest are still being obtained and the range of valuable products is being gradually extended.

As there is no direct link between the names of the products which are placed on the market and the patents in which they are described, scientific papers disclosing the constitution of the former are always of interest. H. Lederer (*Textilber.*, 1931, XII, 461), has recently examined Ciba naphthol R.P. (S.C.I.) and identified it with phenyl- α -4-hydroxynaphthyl ketone (formula annexed).



It will be noticed that it differs from the majority of the naphthols, in that it does not contain an arylamido-group. It is readily ethylated with caustic soda and diethyl sulphate to form 4-ethoxynaphthaphenone. Naphthols of similar structure to the above are described in B.P. 343,016 (Soc. Chem. Ind. in Basle), these being 4-hydroxy-1-naphthoic esters. They couple in position 3, with diazotised amines; aniline gives a red pigment and *m*-aminobenzyl alcohol a red-orange.

Carbazol derivatives have already been used as "arylamides," and a new starting material for these is 5-hydroxy- α -naphthacarbazole-4-carboxylic acid, which

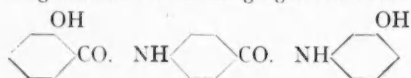


it will be seen is an analogue of β -oxynaphthoic acid, and like the latter, it can be converted to arylamido-derivatives, which can then be coupled with diazotised amines in the usual manner (B.P. 347,113, I.G. Farbenind. A.-G.).

Similar, but slightly less complicated dyes derived from carbazole arylamides, are those of B.P. 343,164 (*idem*), e.g., the brown obtained from the analide with 5-nitro-*o*-anisidine.

"Naphthols" as Arylamides

"Naphthols" can also be made from simpler starting materials, e.g., phenols, especially salicylic acid. As an example, salicylic acid is condensed with *p*-aminobenzoic acid in toluene in presence of phosphorus trichloride at 110°, and the product then condensed with *m*-aminophenol in dimethyl-aniline, using the same condensing agent as before.



to give the compound shown in the above formula (B.P. 340,661, *idem*). The similarity in structure to the conventional naphthols will be observed. Other products of the same type are obtained by condensing 4-arylamino-salicylic acids with arylamines by the usual methods (B.P. 336,428, *idem*). Good affinity for the fibre is obtained in both cases and development can be carried out in the normal manner. The presence of free sulphonic and carboxylic acids is, of course, to be avoided, if fast dyeings are required. It will be seen that these naphthols are arylamides in a double sense, and the same idea can be seen in other recent products. The two arylamido-groups can be joined to the same naphthalene

nucleus as in B.P. 336,800 (*idem*), where a naphthalene-1:4- or 1:5-dicarboxylic dichloride is condensed with two molecules of an aminophenol, such as *m*-aminophenol. Alternatively, they may be both attached to a symmetrical diphenyl residue, such as a dicarboxydiaryl-carbamide, being obtained from this by condensing with two molecules of an aminophenol (B.P. 338,314, *idem*). In the former case the structure will be



and in both, the hydroxyl groups are in different residues from that to which the carboxylic groups are attached, thus differing from naphthol-AS.

Selected Couplings

With so many different bases and salts from which to choose, the possible number of azo dyes which can be obtained is very large, and in order to get the best results, judicious selection is necessary in deciding which combinations are to be recommended. F. M. Rowe has shown (*J. Soc. Dyers and Col.*, 1931, XLVII, 29) that resistance to kier-boiling is a specific property of the coupled colour, and not of the components. Incidentally it may be mentioned here that the paper just cited, together with one in which the same author collaborates with F. H. Jowett (*ibid.*, 163) and a third, previously mentioned in the August issue of THE CHEMICAL AGE *Dyestuffs Monthly Supplement*, gives a full account of very extensive investigations into the subject of the kier-boiling of insoluble azo colours. Going back to the question of selected couplings, some recently patented ones may be mentioned. These include (a) diazotised 4:6-dichloro-*m*-toluidine with 2:3-hydroxynaphthoic-3-chloro-2:4-dimethoxyanilide, which gives Turkey-red shades (B.P. 351,585, I.G. Farbenindustrie A.-G.), (b) diazotised alkyl 5-halogenoanthranilate with 2:3-hydroxynaphthoic α - or β -naphthylamide, which also gives reds (B.P. 319,247, *idem*), and (c) diazotised 6-halogeno-3-amino-1:4:5-trimethylbenzene with a 2:3-hydroxynaphthoic arylamide, which again gives reds (B.P. 348,811, *idem*).

Self-Coupling Arylamides

Instead of coupling a base with a salt, suitable self-coupling arylamides containing amino-groups can be used. The procedure, in dyeing with these, is to treat the substance with nitrous acid on the fibre, and then with alkali or other suitable substance which will cause self-coupling. By this method, 2:3-hydroxynaphthoic-*m*-aminoanilide gives a red (B.P. 347,609, J. F. Turski). The coupling may take place either intermolecularly or intramolecularly. Two bases, which are recommended, are the 3-chloro-4:6-dimethoxy- and 2-chloro-4:5-dimethoxy-anilides of 2:3-hydroxynaphthoic acid (B.P. 343,419, I.G. Farbenindustrie A.-G.). Finally, there may be mentioned the azoxy- or azo compounds, suitable for diazotisation and coupling on the fibre, which are obtained by the reduction of certain compounds containing nitrobenzoylamino-residues, namely, *m*- and *p*-nitrobenzoic hydroxyarylamides, having a free position ortho or para to the hydroxyl group, and aromatic diaminocarboxylic acids, in which at least one amino-group is nitrobenzoylated, or similarly substituted nitro-amino-carboxylic acid (B.P. 337,563 and 337,902, *idem*).

The Future of Rayon

THE high susceptibility of present rayons to physical forces, such as stretching, suggests that a practical use may be discovered for these in improving rayon. Reflecting on the success which has attended the synthetic production of other products, it is impossible to believe but that in the future rayon will be so much improved as to be much more perfect than the older fibres considered from the manufacturer's point of view. It can only displace cotton, wool and silk, in proportion as it equals or becomes superior to these fibres. There is no reason, however, why it should not be made more perfect than these older fibres.

The Viscosity of Cellulose Solutions

An Index of the Extent of Chemical Degradation

A MEETING of the Manchester Section of the Society of Chemical Industry was held at the Engineers' Club, Manchester, on Friday, December 4, when a number of short papers dealing with textile and dyeing problems were read. Dr. T. Callan was in the chair.

Dr. D. A. Clibbens, of the Shirley Institute, Didsbury, Manchester, in his paper on standardised methods for determining the rate of flow of cotton in cuprammonium solution stated that the viscosity of a cellulose solution was a very sensitive index of the extent of chemical degradation of the dissolved cellulose. In most cellulose using industries the extent of such chemical degradations was a very important factor, either in determining the proper running of a process or the proper performance of the product. For this reason, cellulose viscosity measurement was being used to an increasing extent as a method of works control, process control or of material specification. The industries in which the viscosity measurement had already proved its value were the cotton, linen and rayon industries, the explosives industry (nitro-cellulose) and the pulp and paper industry. The rayon manufacturing industry had a control or specification of the raw material, and so had the celluloid, cellulose varnish and lacquer industries.

Importance of Viscosity Measurement

It was, however, in the textile industry that the closest attention had been paid to the possibilities of the method, and most of the data enabling its technical value to be assessed were derived from textile investigations. The importance of the viscosity measurement in the textile industry was due chiefly to the fact that increasing chemical degradation of the cellulose was accompanied by a steadily increasing fall in the tensile strength of the cellulose fibre which went hand in hand with the fall in the viscosity of the cellulose in solution. Measurement of viscosity gave some kind of absolute measure of the tensile deterioration of cellulose textiles due purely to chemical process, as, for example, to the bleaching operation. It separated those tensile effects which were due to chemical alteration of the cellulose from those which were due to purely mechanical causes such as the structure of yarns or fabrics.

The fibrils of the cotton hair could only be regarded as large aggregations of the ultimate cellulose unit. The geometrical form of the fibre itself, and of its fibrils, could only be regarded as a reflection of the form of the ultimate cellulose unit which was of a relatively very long and thin structure, or, so to speak, a molecular chain built up from a large number of glucose groups strung end to end. The tensile strength of the fibre was determined by the intactness of these molecular chains. The chemical attack of the cellulose which produced loss in strength of the fibre caused a chemical fission through hydrolysis or oxidation of the molecular chains which could be regarded as the direct explanation of the loss in strength. When the cellulose was dissolved in suitable solvents this chain-like structure was preserved and caused a high internal resistance to flow of the solution owing to the entanglement of chain on chain as they slipped past one another. The chemical attack of the cellulose, which was in effect a shortening of the ultimate units or chains, lowered the resistance to flow, that was lowered the viscosity of the solution. These views, gave, at least, a mentally satisfying picture of the reason for the close correlation of the viscosity of cellulose in solution and the strength of the cellulose in the fibre on which the important application of viscosity in the textile industry depended.

Difficulties Associated with Measurement of Viscosity

The measurement of the viscosity of cellulose was accompanied by difficulties which were not encountered in ordinary viscosity measurements, almost the only solvent suitable for the purpose being cuprammonium. When cellulose was dissolved in cuprammonium it was very easily oxidised at the ordinary temperature by the oxygen of the air, which oxidation, like any other form of attack, induced a viscosity of the solution. The preparation of the solution and the origin of the viscosity had to be carried out substantially in the associated with the colloidal nature of cellulose. Therefore,

any general method of measuring cellulose viscosity had to be absent of air. Other difficulties of measurement were deal with a very wide range of viscosities. The high viscosities of cellulose solutions were abnormal in their flow properties. Their viscosity appeared to diminish as the driving pressure under which the solution flowed was increased. The result was that the viscosity of measurement had no strict physical significance. It had a practical significance only when the conditions of flow were standardised.

Catalytic Tendering by Dyestuffs

In his paper on the occurrence and detection of certain faults on textile fabrics, Mr. O. S. Rhodes stated that during the examination of complaints on guaranteed fabrics, both from retailers and actual users of the various materials, there were naturally found to be some which were due to unsuspected defects in either the dye, the fabric, or one of the processes through which the cloth passed during manufacture. If the cause of such defects could be found the defect could in most cases be eventually eliminated or reduced to a negligible quantity. He therefore wished to make a plea for the greater use of what he might almost call photographic methods of detection of damage due to oxycellulose formation. It was always of the greatest importance to show the location and distribution of the damage, and for the purpose a test which stained the damaged parts much more deeply than the rest of the fabrics was most necessary. For instance, the oxycellulose test was of the utmost help when the almost classic case was discovered of catalytic tendering of cotton by Grelanene Red 2B when overbleached. In illustrating this he exhibited a piece of bleached handkerchief fabric containing yarns dyed with Grelanene Red 2B and Duranthrene Orange 4R which had been subjected to the oxycellulose test. The very much deeper black deposit upon the Grelanene Red 2B dyed yarn showed how seriously the yarn had been tendered as compared with the almost complete freedom from degradation of the Duranthrene Orange 4R dyed yarns.

Other papers were read on an empirical classification of vat dyestuffs (C. M. Whittaker), difficulties in the quantitative measurement of dyestuffs adsorption (S. M. Neale), and the skin effect on viscose rayon (J. M. Preston).

Scottish Dyestuffs Market

THE recent anti-dumping duties imposed under the Abnormal Importations Act have given satisfaction in Scotland, particularly in Dundee, the centre of the jute trade, and in Dunfermline, famous for its linen. Both of these towns have suffered considerably from the effects of foreign competition. It is also considered that the woollen and worsted industries at Alloa will feel the advantage of the new Order. An old Glasgow industry of bottle making may also be revived as a result, this industry having been almost extinguished by foreign competition. Although employment in the Tweed mills at Hawick and elsewhere in the South of Scotland has been better these last few weeks, there is still room for considerable improvement as manufacturers are by no means working up to full capacity, although there are one or two exceptions. There is still considerable caution displayed in giving orders on the new 1932-33 winter season samples, and as is often the case nowadays the more novel effects are bringing in the larger orders. Dyers remain only moderately well employed, but dyestuff orders are satisfactory.

North British Artificial Silk Proposals

IN a circular letter to the shareholders of the North British Artificial Silk Ltd., Mr. Ernest Walls, the chairman, recalls that at the recent meeting he foreshadowed proposals for the reduction of the capital. Extraordinary meetings will be held on January 14 to give effect to the proposals, under which the Preferred Ordinary and Deferred shares will be amalgamated in one class of Ordinary share and the total capital written down from £385,000 to £102,500. This will be done by cancelling 10s. per share on the Preferred Ordinary shares and 6d. per share on the Deferred shares, and consolidating every 20 Deferred shares of 6d. into one share of 10s. The capital will then again be increased to £385,000 by the creation of 385,000 new 10s. shares. The name of the company is to be changed to North British Rayon, Ltd.

Monthly Metallurgical Section

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NOTICE.—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

Surface Treatment of Aluminium and its Alloys

A Survey of Modern Practice

The following article, which is based on information supplied by the British Aluminium Co., Ltd., of London, deals with the principles of surface treatment for various purposes; protection against chemical, physical and mechanical action; electric insulation; metallic contact; and heat absorption.

PROTECTION is an important problem in the surface treatment of aluminium, whose characteristic properties are such as to make many of the usual methods either difficult to apply or impossible. With regard to protection from mechanical injury, from the point of view of surface treatment this is largely confined to reduction of frictional wear. In this connection two interesting examples are the use of zinc plating to prevent aluminium parts from seizing and the recent introduction of a soft rubber surface coating to reduce abrasion. Protection from physical injury is again, from the point of view of surface treatment, limited in practice to providing a surface relatively indifferent to temperature changes. For high temperature resistance the oxidation processes provide a satisfactory treatment for aluminium. The question of protection from chemical effect, or corrosion, is, however, by far the greatest problem which protective surface treatment is called upon to deal with. Corrosive agents may be natural corrosive agents, such as the atmosphere, moisture, sea water, etc., or industrial corrosive agents, as met with by metals in industrial applications. In the latter case the conditions of corrosion may be said to be stable; but in the former case the conditions are by no means constant, and the surface treatment has to be capable of responding to the differing conditions which are encountered.

Essentials of Surface Coatings

The first essential of any surface coating from the point of view of chemical protection is, therefore, that it should be a continuous envelope sealing completely the metal which it protects. The corrosion attack is then limited to the material of the coating, and since that material can be specifically chosen to be inert for the particular conditions, it can be reduced to a minimum. If the coating is not a completely enveloping surface, that is to say, if it is porous, the corrosive effect will then depend on whether the coating is a conductor electrically. If it is not, the rate of corrosion of the basis metal is determined by the rate of access of the corrosive agent, but if it is a conductor, electro-chemical action comes into play and the character and intensity of corrosion effect depend on four factors: (a) the relative solution pressures of the basis and coating metals, (b) the nature of the electrolyte, (c) the nature of the anodic and cathodic corrosion products, and (d) the rate of access of oxygen.

Certain differences, however, distinguish aluminium from other metals where surface treatment is concerned. Its mechanical qualities which allow of obtaining a smoother surface than most metals, especially ferrous metals, are advantageous in mechanical surface treatment. Its chemical properties, outstandingly its extreme oxidisability, high position in the electro-motive scale and its high reactivity, on the other hand, place it at a disadvantage for all processes involving chemical reaction, and these difficulties have to be overcome by special methods applicable to the class of phenomena entering into the particular process. Common to all methods of surface treatment is the necessity for a *clean basis metal surface*, and in the case of aluminium this is a particularly difficult problem on account of the chemical reactivity referred to, and much research has been carried out

on the subject. The general method of pre-cleaning depends on, first, a degreasing of the surface with an organic solvent, and, second, an attempt at oxide removal by both alkali and acid pickles, for which a vast number of formulae exist. For pure aluminium, chiefly hydrochloric or hydrofluoric acid, or mixtures containing the latter, or solutions of sodium salts, are used; for commercial aluminium and alloys nitric acid is generally used.

Mechanical Surface Treatment

British patent 209,063 was taken out in 1924 to cover a process for pressure plating or mechanically coating aluminium with a heavy metal such as copper, lead, silver, nickel, etc. This process consisted in heating the aluminium, cleaning the surface with special compounds, continuing the heating to 300–550° C., spreading the layer of metal, such as tin, on the face of the aluminium and pressing on a thin cleaned sheet of the heavy metal, using hydraulic pressure, and cooling under pressure. A mechanical process of pressure plating is also made use of in the German Allautal and Duralplat and the American Alclad alloys, the high tensile strength alloys Lualal and Duralumin being plated by a special pressure process with a considerable plate of pure aluminium, which renders the alloys corrosion resistant.

Polishing is rather more difficult in the case of aluminium than in the case of other metals, principally on account of its softness. Softer polishing wheels and finer abrasives are, therefore, necessary to prevent the metal surface being dragged out and the wheel becoming glazed. The high thermic conductivity and specific heat are also disadvantages, particularly as the coefficient of friction is high and the heat developed by rubbing is, therefore, transmitted rapidly through the articles, making handling difficult. In addition there is the risk of the polishing wheel burning. The lightness of the metal, however, facilitates manipulation. The technique of polishing to give a high mirror-like polish involves three stages after the preliminary cleaning operation: Smoothing down the surface (especially in the case of sand casting), buffing, and rubbing up with a polishing paste. Surface smoothing is accomplished with emery wheels the surface of which is kept thoroughly lubricated. Buffing is carried out with soft buffing wheels of chromed leather, felt, rag or calico, using some abrasive powder mixed with a lubricating grease, and the final rubbing up is done with a suitable metal polishing paste such as "Almin." If a less brilliant surface is considered sufficient, burnishing is substituted for polishing. Here, after pre-cleaning, the articles are placed in a tumbling barrel containing small steel shot in a soap solution and revolved at a high speed, which produces a continuous rolling motion of the shot. On removal from the burnishing barrel the articles are immediately placed in a second revolving barrel containing hot sawdust to dry and brighten them.

Ornamental finishes are obtained by scratch-brushing, a fine satin finish being given to aluminium by a scratch brush running at 1,400 r.p.m., the wheel being run dry with a little lime used on the wires to keep them free from oil or grease. Curling, which consists in rubbing the surface over in circles with a cork or tool covered with leather and coated with fine emery, gives a pseudo "engine-turned" surface.

"Metallisation"

The spray coating or "metallisation" of aluminium is usually confined to galvanising or coppering, the former either for protective reasons or as a preliminary to gilding or plating with other metals, the latter for decorative purposes. The zinc or copper is applied with a Schoop "metal pistol": a wire of the metal is automatically fed through the pistol body into the nozzle where it meets the hot zone of an oxy-blowpipe flame which is surrounded by a cone of compressed air; the metal as soon as it becomes molten is atomised and forced at high velocity against the surface of the aluminium, which is, as usual, pre-cleaned and sandblasted.

Surface Protection by Chemical Methods

A "matt" or frosted finish on aluminium is produced by dipping the metal first in an alkaline and then in an acid pickle, the latter being necessary to remove the black deposit which the alkaline dip generally produces. The alkaline pickle is usually caustic soda of strength not less than 10 per cent. by weight, at about a temperature of 95° C. Two cold water rinses are necessary before dipping in the acid bath, which consists of a mixture of acids, sulphuric and phosphoric (9:1)—about 2 per cent. by weight—or in cases where the black scum is not readily removed, sulphuric and nitric (1:1) of greater strength. The metal has then to be thoroughly rinsed, first with cold and then with hot water, and dried with hot sawdust.

A dead white coating can be given to aluminium by boiling it in "milk of lime" which contains a little calcium sulphate, and subsequently baking the treated metal. This coating adheres very firmly and does not remove on bending, it cannot be rubbed off by the fingers, but a pencil eraser removes it. The texture is extremely fine-grained, and the surface can be enamelled or painted or sized if required for ink. Various baths have been compounded to give a black finish, some of the solutions being very complex. In some cases the surface is treated with copper ammonium chloride and heated to produce copper oxide, or is subsequently treated with sodium sulphide or lamp black is rubbed in. In another case bath treatment is given with tanning and the metal is heated gently, when dry a dull black finish is imparted to it. After an acid dip in 3:1 sulphuric acid, the metal is treated with a mixture consisting of 150 grams antimony bichloride, 100 grams manganese nitrate, 20 grams graphite and 250 grams hydrochloric acid in 1 litre 90 per cent. alcohol. It is then coated with a varnish made up of 50 grams sandarac and 100 grams nigrosin in 1 litre of spirit.

The Jirotko process for producing a coloured film on aluminium by immersion in a bath of an aqueous solution of chromates, etc., and similar salts, has been in use for some time, but recently British Patent 338,204 provides a modification, the bath consisting of chromates or dichromates or both in alkaline solution, with or without additions of metallic salts. Here the action of the alkali is to dissolve away the aluminium to a certain depth, the chromate then producing a uniform dispersion of the alloying constituent exposed.

(To be continued)

A New Creep-Resisting Steel

At the present time the question of creep-resisting steel is in such a state of flux that no standard specifications have been drawn up. The only step which has been taken in this direction is the drafting of a British Standard Specification (No. 10, Part 4—1931) for pipe flanges, for working steam pressures above 600 lbs. and up to 900 lbs. per square inch, and temperatures up to 800° F., where particulars have been drawn up for stud bolts for use at temperatures over 750° F. "Durehete," a new product of Samuel Fox and Co., Ltd., who are associated with the United Steel Companies, Ltd., of Sheffield, is now claimed to meet this specification entirely, in its physical properties, and is said to be much superior to the nickel-chrome-molybdenum type of steel which is also referred to in the specification, as unlike nickel-chrome-molybdenum steel it is almost immune from embrittlement under repeated heating and cooling. Tests carried out at the National Physical Laboratory have shown that this new steel has a creep rate of less than 10⁻⁷ inch per hour, after a period of 20 days at a temperature 480° C., under applied stresses of 5 to 10 tons per square inch.

Reorganisation of Steel Industry Confederation Campaign in Tees-side Area

IN pursuance of its policy to secure the widest possible support for its proposals for reorganising the industry, the Iron and Steel Trades Confederation has recently held a number of meetings in the Tees-side area. Mr. Arthur Pugh, general secretary of the Confederation, was the principal speaker at South Bank, Brotton, Stockton, and West Hartlepool. Other speakers included Mr. W. Mansfield, M.P.; Mr. James Walker, M.P.; Mr. F. F. Riley, M.P., and Mr. J. Herriotts, M.P.

In the course of his speech Mr. Pugh said the proposals of the Confederation consist mainly of the reorganisation of the industry by the establishment of a public utility corporation, and if this does not solve their difficulties, the application of a tariff. Unless we could find the necessary leadership and capacity for reorganisation, the iron and steel industry of this country would be unable to meet the changed world conditions. It was essential that the spirit of defeatism, so prevalent in the ranks of those at present controlling the industry, should be removed. Our continental competitors complained that they could not find anyone in a position of authority to speak for the whole industry, and with whom they could negotiate. If, then, the iron and steel industry was properly organised, this would be possible. The Confederation's scheme, declared Mr. Pugh, was not nationalisation in the ordinary sense. It was really the community undertaking what private enterprise had failed to do—the organisation of a great basic industry for the public good.

A resolution supporting the Confederation's proposals was carried at each of the meetings.

United States Steel Merger

ACCORDING to the *New York Evening Post*, an agreement ending the litigation of the minority stockholders of the Youngstown Sheet and Tube Corporation against the thousand million dollar merger with the Bethlehem Steel Corporation has been reached tentatively. The actual negotiations for a merger of the Youngstown Sheet and Tube Corporation and the Bethlehem Steel Corporation were concluded more than a year ago and were ratified by the majority of the stockholders, but a large minority of the stockholders, headed by Mr. Cyrus S. Eaton, the well-known banker and industrialist, protested and appealed against the decision in the Ohio Court. The merger was thus defeated, but it is now believed that the tentative settlement will open the way to a new consolidation involving other large Mid-Western steel companies, such as the Republic Steel and the Gulf State Steel

Platinum Production in Canada

ACCORDING to figures issued by the Dominion Bureau of Statistics at Ottawa, 68,116 fine ounces of platinum, palladium, rhodium and other metals valued at \$2,439,128 were produced from Canadian mineral ores in 1930, as compared with the 1929 total of 29,837 fine ounces, worth \$1,656,045. During the year, 19,835 ounces of platinum contained in concentrates, and 285 ounces of scrap were exported, making a total of 20,120 ounces, valued at \$1,626,598, as against 2,910 ounces valued at \$225,519 during the previous year. Electrolytic and sponge platinum metals are now being utilised in the manufacture of iridium-platinum-palladium jewellery alloys, electrical instruments, high temperature thermocouples, vacuum tube amplifiers, heating elements, electro-plated table ware, chemical laboratory ware, dental supplies, and as catalysts for chemical processes.

German Sponge Iron

It is announced that the Eisenschwamm Co., which was formed by Krupps and the Vereinigte Stahlwerke to develop the Norsk-Staal process of making sponge iron, has been operating its plant during the early months of 1931 with results which have come up to expectations and have established the principle of the process. In view, however, of the continued depression in trade, Krupps and the Vereinigte Stahlwerke have decided for the time being to close down the plant, but it is hoped to restart operations immediately economic conditions show an improvement.

Metallurgical Topics: Monthly Notes and Comments

From Our Own Correspondents

Modern Research on "Pure" Metals

A NOTABLE feature of metallurgical research during the past few years, to which the transactions of the various metallurgical societies and institutions have made important contributions, and which have likewise engaged the attention of the principal research associations devoted to metallurgy, has been the preparation of pure metals, not as an end in itself, but as an aid to the investigation of the influence of impurities on metals. During the past sessions of the leading societies this trend of modern research has been very greatly in evidence, while the work of such institutions as the National Physical Laboratory, the Research Department of the Royal Arsenal, Woolwich, and of the laboratories of some of the Universities specialised for this kind of work, has borne very largely upon research of this nature. Of course, the investigations of the influence of impurities has been going on for years—a century or so at least—but until quite recent years it was carried on in an empirical and necessarily inconclusive way. The preparation of pure metals has only been possible with the means introduced comparatively recently; the perfect control of temperatures; the intensive study of non-contaminating refractories, and the use of the high-frequency induction furnace which agitates and scours its melt in a non-contaminating atmosphere and so secures conditions not formerly attainable. Earlier research work on the influence of any one "impurity" was complicated and its results often hopelessly masked by the presence of other impurities. To isolate the effect of any one of them was a task which, in the circumstances, was beyond achievement.

Grades of Purity

METALS described as "commercially pure," "chemically pure," and "spectroscopically pure" are now procurable, and the distinction between their value in research properly understood. The very faintest traces of certain other constituents will, as is now better known than ever, alter almost fundamentally the properties of a spectroscopically pure metal, such, for instance, as the ultra pure New Jersey zinc, and only when such a base is employed, with all possible precautions against contamination with any other metal can the true effect of an added constituent be rightly observed and accurately evaluated. Thereafter controllable alloys of all kinds can be built up, on condition that additional constituents are likewise pure. Hitherto, the conception of what is pure has been vague, but the noteworthy feature of recent work has shown that the word purity must be, in metallurgical research, narrowed to its uttermost limit. When using commercially pure base metal and commercially pure metallic, or non-metallic "additions," the results still lack precision, and remain empirical. Purity, in the last resource, means spectroscopical purity. In all other works, traces of unknown and quite infinitesimal impurities may vitiate the conclusions. Modern metallurgy is tending in the direction of an exact science and some day we shall be able to assign an exact numerical constant to the influence, on any given property of any given alloy, of the addition of a given amount of any particular constituent. It will be exceedingly interesting to watch this development, and it is, even now, possible to speculate as to whether the "constant" to be assigned to each element, in an alloy, is a function of any known constant, such, for example, as the atomic weight, the atomic number, or the atomic volume, of the elements entering into its composition.

The Institute of British Foundrymen

THE Institute of British Foundrymen, which has just concluded its twenty-eighth annual Conference, is a highly specialised institution which has had a remarkable record of progress and useful work. Its membership, at the end of last year, was 1,844, and it has a dozen very live branches. While concerned chiefly with iron founding, it has, of recent years, devoted increasing attention to foundry work in non-ferrous metals and alloys, and the proportion of papers read at its general meetings and before the branches on these subjects is steadily growing. There is some degree of inevit-

able overlapping with the Iron and Steel Institute and with the Institute of Metals, but it is of a nature to which little or no exception need be taken, as the work of the Institute of British Foundrymen lies rather in the application to severely practical ends of the more purely scientific research work undertaken by the two other leading metallurgical institutions. Another exceedingly useful aspect of the work of the "I.B.F." is the close relationship it maintains with American and Continental foundry associations such as the French Foundrymen's Association and the American Foundrymen's Association, relations which materialise, at the annual meetings of all three, in many valuable "exchange papers" by members of each of these institutions. The American "exchange paper" read at the recent meeting in Birmingham was a masterly contribution entitled "Some Considerations and Tests for Cast Materials for High Temperature, High Pressure Service," by L. W. Spring, the chief chemist and metallurgist of the Crane Co., Chicago.

Metals for High-Temperature and Pressure

THIS subject is one of supreme importance in modern engineering, and the foundry is faced with the competition of wrought materials of all kinds which render such a paper, written on the subject of cast metals, exceedingly timely. Power station temperatures and pressures are rising enormously, while in some oil-cracking and chemical-process work the former attain as high as 980° C., and pressures up to 3,100 lb. per sq. in. have been tried. Incidentally, it may be mentioned that in the Loeffler boilers, as employed at Vitkovice, and elsewhere, temperatures of 932° C. are actually combined with pressures of 1,568 lb. per sq. in., and it is understood that molybdenum steel is used where these conditions have to be met. Mr. Spring, in his paper, limits himself to castings, but discusses both ferrous and non-ferrous castings in this connection. In respect of the former, chromium-molybdenum cast steel tests show that this material stands up well to the creep conditions materials may be called upon to withstand in severely high-temperature high-pressure work, and the effect, in particular, of molybdenum, is discussed. Steel castings are, indeed, awarded pride of place for such work while, if not for strength, for corrosion under severe service conditions, the chromium-nickel alloys render good service. The paper is an excellent one, but does not lend itself to abstracting. Another very excellent paper, reviewing many aspects of the properties, constitution and applications of cast iron, was contributed by J. G. Pearce, the Director of the British Cast Iron Research Association. Mr. Pearce gives an interesting account of the "germ" theory of graphite formation in cast iron and shows how the precipitation of finely disseminated graphite may be controlled in foundry operations. Finally, mention should be made of an excellent paper by J. Arnott on silicon as an alloying element. Hitherto regarded chiefly as a deoxidiser, Mr. Arnott deals with its use as an actual alloy constituent and holds a special brief for silicon-bronze, which he claims to be in some respects superior to gun metal, and quite as resistant to many corrosive influences as that metal.

Researches on Lead and Lead Alloys

AMONG various researches which are in progress under the British Non-Ferrous Metals Research Association, that on the properties of lead and some of its alloys deserves special mention. The initial object of this research is to ascertain the causes of the failure of lead sheathing of electric cables which exhibited intercrystalline cracks, but its scope has been extended to include a study of the mechanical properties of lead and its alloys. By this extension, not only is the original objective to be more quickly attained than would otherwise have been possible, but improved materials have also been discovered and the relative strength properties of lead and a number of alloys, both already known and newly discovered, have been established. Properties already investigated included elasticity, resistance to stress, compressibility at raised temperatures, and more recently rolling and annealing. The causes of the cracking of lead cable sheathing have been

established and the relative superiority of a fairly wide range of alloys has been demonstrated. Certain ternary alloys have been found particularly valuable, have been patented, and are now being widely used for the sheathing of electric cables. Apart from this, mechanical strength at low and elevated temperatures, and the effect of prolonged and intermittent stress, have also been investigated, as well as the effect of certain impurities and of intentional alloying additions and of corroding media. In addition, the compressibility of lead and its alloys in relation to temperature has yielded information of value in the manufacture of these materials in the form of cable sheathing and pipe.

Age-Hardening of Austenitic Ni-Mn Steels

EXPERIMENTS on the applicability of age-hardening to austenitic non-magnetic steels are reported in Technical Publication No. 419, of the American Institute of Mining and Metallurgical Engineers. These experiments have been carried out on nickel-manganese steels containing (1) titanium 1.9-2.0 per cent., and (2) molybdenum 1.8-2.3.5 per cent.; the basis alloy selected containing nickel 15 per cent., manganese 10 per cent., and very low carbon. The published report, which is by F. R. Hensel, embodies detailed data, from which it is concluded that the addition of 3-5 per cent. titanium or of more than 1.3 per cent. of molybdenum to a ferrous nickel-manganese austenitic alloy makes it possible to obtain marked precipitation hardening. Both series of age-hardened alloys remained non-magnetic throughout heat- and mechanical treatments. In the titanium series, ageing was accompanied by pronounced shrinkage, but after ageing, at 600° C., to maximum hardness, the titanium alloys presented a troostitic appearance, and X-ray analysis showed that the lattice constant changed during ageing. The tensile and elastic properties of these titanium alloys, moreover, were increased by the precipitation hardening to a degree which renders them of interest from the engineering point of view. Electrical resistivity showed an abnormal change in that it first increased and subsequently decreased. Ageing is also pronounced to have an unfavourable effect on the tensile properties of the molybdenum series, as elastic limit falls, and the alloys lose their ductility without corresponding increase in mechanical strength.

Extended Uses for Aluminium

ACCORDING to a report issued by the United States Bureau of Mines, certain of the established uses for aluminium showed decreases in 1930 in demand for the metal, but these were more than offset by gains in other directions, particularly in strong aluminium alloy materials, in electrical conductors, and in the use of aluminium for paint. Lessened consumption was to be noticed especially in the automobile and aircraft industry. Builders of truck and bus bodies, however, increased their demand by incorporating aluminium strength-members in body frames, in addition to low-stressed parts, such as panelling and roofing. Trucks for heavy commodities, such as coal and sand, and light merchandise have also utilised aluminium in the form of strong alloy sheets and structural shapes, heat-treated to develop maximum physical properties. Strong aluminium alloy wire was introduced by a prominent fence manufacturer as a new material for woven fence and barbed wire. Another manufacturer started the production of aluminium window screening. In both instances durability was the prime advantage claimed; this resistance of aluminium to atmospheric corrosion was also responsible for a steady increase in the use of the metal as a roofing and sheet metal material, and for an even sharper increase in its use as an ornamental metal on the exteriors of commercial and public buildings. This latter field showed great activity in 1930. Aluminium spandrels, window frames and sills, sky-lights, finials, grilles, balustrades, store-fronts, and other ornamental metal applications, both exterior and interior, all satisfy the architect's demand for a durable white metal that is strong and yet easily fabricated. The consumption of the metal in aluminium cable accounts for about 16 per cent. of the United States production. Sales during 1930 were largely in the form of bare stranded cable with a core of steel for transmission lines. During the year, however, a complete line of insulated aluminium wires and cables was launched by a large manufacturer as a supplement to similar products of copper. This development extends

the use of aluminium to include insulated cables for underground network systems, automobile starter cable, aviation, and automobile wiring systems, and other wire and cable products. Electroplating on aluminium, and chemical or electro-chemical processes of colouring aluminium has also been commercialised; these finishes are extending the use of the metal by providing a change or modification of its characteristic colour. Heavy aluminium foil or thin sheets of aluminium have been found to offer a protective coating for oil tank cars, pipe-lines, storage tanks, and similar structures. This aluminium foil, applied like wall paper with proper adhesives, is used to protect the underlying surface against sulphur fumes or general atmospheric corrosion, and to furnish a radiant-heat reflecting surface.

The Non-Ferrous Metals Market

THE general situation in non-ferrous metals at the opening of this week was strengthened by the important news from Washington concerning War Debts. This confirmed the hope entertained that something would be done to remedy the European economic crisis and general trade depression. Vital developments now appear to be pending which are likely to make for a return of confidence, and thus hasten a trade recovery, which is already overdue. The general financial outlook having been materially strengthened, this should undoubtedly facilitate a world industrial revival, and prospects in the non-ferrous metals, as in other commodities, are brightening up; while increased trade-absorbing capacity can be reckoned with. According to the *Financial News*, prices in all directions moved rapidly upward, even in the case of copper, the position of which in the past week weakened considerably further under a reduced consumption and statistical conditions worse than ever before. There is now a possibility of an enlarged outlet in the manufacturing trades, and also in the electrical industry, where the placing of new schemes may become accelerated. This brighter aspect, and the remarkable buoyancy which has characterised the American and European Stock markets, precipitated a sharp revival of speculative activity in the metal early this week, resulting in prices being materially lifted from the new low record touched. Dealings in standard copper have been on a considerably heavier scale, and prices rose steadily, eventually reaching £38 7s. 6d. for three months' delivery. At this level there has, therefore, been a recovery of well over £4 per ton from the record low level touched for all times near last week's close. Pig lead and zinc were also very conspicuous in the recent rallying movement. Their appreciation indeed, was relatively greater than in other metals, in spite of the fact that, in the case of pig lead, there has been no improvement in the statistics. G.O.B. zinc, which had dropped early in the month to £9 13s. 9d., has been lifted latterly to £12 16s. 3d., thus denoting an improvement of well over £3 per ton. This figure, however, is still at a few pounds below cost of production, which has tended to encourage dealers' support and to stimulate trade buying, with some little improvement shown in the consuming trades, particularly galvanised sheets.

Resisting Corrosion by Sea Water

THE extraordinary corrosion of metals by contaminated waters, especially sea waters close in shore, is the subject of a new booklet on "sheathing metals," which has been published by I.C.I. Metals, Ltd. The protection of wood exposed to the action of sea water was first effected in 1761 by the use of copper. Subsequently cheaper substitutes appeared and in 1832 Muntz metal was introduced as a patent alloy which could be used not only for sheathing but as a coating for wooden piers and wharves in tropical and semi-tropical waters where attack by marine borers was also to be resisted. There are, however, certain localities where water conditions are such that even Muntz metal is unsatisfactory, and in consequence, new alloys have been developed, the principal of which are elio- and cupro-nickel. The former is strongly recommended where water conditions are known to be abnormally severe; the latter is suitable for use under all conditions, but by reason of its greater strength and its apparent non-corrodability, it can be used in thinner gauges. Copies of this booklet may be obtained on request from Imperial Chemical House, Millbank, S.W.1.

Trade, Commerce, Finance : The Month in Review

From Our Market Correspondent

DURING the past month the iron and steel trade has continued to mark slow time, no events of any particular importance having taken place. It almost goes without saying that there is still an entire absence of any development which might be construed as an improvement in trading conditions. The anxious desire for such a change has, in many instances, led one to put a rosier complexion on minor events than their actual importance warranted, but these have too often failed to fulfil the hopes that they have been allowed to arouse; and the industry is now labouring under a depression which will be the harder to shake off the longer it lasts.

There is no quarter to which one can turn for inspiration. Little comfort can be found in the trend of political matters. Time after time the possibility has arisen of the overthrow of the present Government, but on each occasion they have managed to avert the crisis, and notwithstanding the marked fall in the Labour vote at the by-elections, the Conservative promise of safeguarding is still a very remote prospect. There is no further official information available as to the progress of the Government's scheme for interfering in the control of the iron and steel industry, but it is certain that they are finding the matter not at all easy to handle. The financial groups which are behind the scheme are, we believe, not showing complete unanimity. Their commitments in the iron and steel trade are so extensive as to arouse some apprehension, and something akin to jealousy among them. If the proposed scheme is carried through they will be bound to make considerable sacrifices which may be rather unevenly distributed and that is no doubt a source of difficulty in the negotiations. After all, there need be no sympathy wasted on the banks, particularly since the Big Five came into being. It is the considered opinion of many who are competent to judge, that the grouping of the banks into these few large units has been inimical to industry generally. They have largely done away with that more or less intimate connection between the industrialist and his banker which played a great part in the development of industry; and they have ill-advisedly financed huge schemes which were practically foredoomed to failure.

Competition in the Steel Trade

In the steel trade itself, they have in more than one instance fostered one of the great evils from which the trade is suffering, namely, the cut-throat competition from concerns which at the time were in a hopelessly insolvent position. To such undertakings, money has been freely advanced, only to be squandered in this mad attempt to wrest a larger share of the available business from the other works. Not only that, but when the inevitable crisis has arisen, the banks again have made it possible for a financial reorganisation to be carried through, the net result of which is to give these undertakings a fresh lease of life so that they can continue the very process which has brought them to bankruptcy. In some of these cases, the waiving of interest on overdrafts over a certain period has amounted to a direct subsidy of a very large amount, whilst no consideration has been shown to the more prudently managed concerns which have managed to preserve their stability, other than allowing them the privilege of paying a high rate of interest for whatever overdraft they may require. However, the long-continued depression is forcing matters to a head, and we are now waiting to see what will be the next move of the financial institutions who hold the power over such a large proportion of the iron and steel industry.

Safeguarding an Urgent Necessity

The recent speech of the chairman of the Consett Iron Co. at the annual meeting of shareholders was of interest, as it was a useful addition to the authoritative statements that have in recent times been publicly made by leaders of the industry. His views on safeguarding, rationalisation and amalgamation, and the general efficiency of the steel works confirm what has been said so many times in other places. Briefly stated they are that safeguarding is an urgent necessity, that rationalisation and amalgamation, while advisable in certain cases, is not a cure for the troubles from which we are

suffering, and that in general, the efficiency of our steel works compares very favourably with that of any other country. One would think that the repeated expression of the same opinion by men whose intimate connection with the industry entitles them to be heard with respect would have more weight with those in high places whose province it is to apply the remedies that lie in their power, but that is not likely so long as the business interests of the nation are sacrificed to political opportunism. It is an irony of the situation that the Iron and Steel Trades Confederation, representing the men employed in the steel works, had to make an official statement contradicting the public utterance of the head of their own Government, and confirming that, as a whole, the British steel works are equal to any in the world in all round efficiency and equipment. The will and the ability to run the industry successfully are also there, but the handicaps, political, social and economical, under which the industry is labouring and over which it has no control, cannot be overcome by any efforts that can possibly be made inside the industry, and the sooner we get a Government that will recognise this truth and act accordingly, the sooner we shall get back on the road to prosperity; but that road is going to be a long and tiring one.

Demand for Reduction in Steel Prices

Another addition to the difficulties of the steel makers is foreshadowed by the official demand which has been made by the Shipbuilding Employers Federation for a reduction in the price of British steel. Their contention is that the price of steel plates and sections at home is so much in advance of continental prices, even after allowing the rebate, that the shipbuilders using British steel are unable to compete with their foreign rivals, and the result is that the tonnage under construction is steadily declining. The threat is that unless the steel makers make an attempt to compete with the continental prices, there will be no alternative left to the shipbuilders but to buy foreign steel. While sympathising with them, it is not unreasonable to ask why they should seek to blame their troubles on to the steel makers. The shipping trade is suffering, like every other, from the general depression, and there are many factors besides the price of steel which enter into the matter. It is not the slightest use expecting the steel makers to take upon themselves the task of rehabilitating the shipbuilding industry. It simply cannot be done that way. If we were to suppose for a moment that the prices of British steel were reduced to such a level as would keep out the foreign steel, it is true the shipbuilders would at once be able to increase their tonnage, but they would do so at the expense of the steel industry which would very quickly collapse in utter ruin, and the last position would be worse than the first. The present trouble for the steel makers is that this demand from the shipbuilders will have its effect at the July meeting of the steel associations, and may be the decisive factor in causing a reduction in the controlled prices, which would be a very serious blow to the steel trade and might well bring final disaster to some of the works.

Possible Effect of War Debts Moratorium

Market conditions remain much the same as they were a month ago. The gradual fall in the prices of pig iron and uncontrolled classes of steel has had no material effect on the weight of business placed, indeed, the shortage of orders is daily becoming more serious. There has been a stiffening in continental prices which may have a beneficial reaction on our market, and the American proposal in relation to war debts, if put into effect, may bring a more optimistic tone to industry and restore some of the confidence which is so sadly lacking. It may be possible, as a result of this, to give a somewhat more cheerful report next month.

There was a slight improvement in the May production figures, which is only natural, seeing that April included the long Easter stoppage. The output of pig iron amounted to 346,500 tons compared with 323,200 tons in April, two more furnaces being in blast over the number in April. The output of steel was 435,100 tons compared with 397,400 tons in April. The output in May last year was 691,900 tons.

Some Inventions of the Month

By Our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature published weekly in THE CHEMICAL AGE.

Alloys

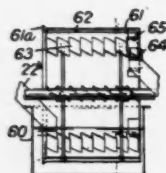
A NON-OXIDISABLE alloy more especially for use in making valves and other apparatus exposed to steam is described in Specification 346,226, International Convention date June 21, 1929, by C. Contal, of St. Cloud, Seine-et-Oise, France. It is composed of nickel (40–50 per cent.), copper (30–40 per cent.), manganese (10–20 per cent.), antimony (5–15 per cent.), and tin (1–10 per cent.) with or without a small amount (up to 1 per cent.) of lead for softening purposes, and is preferably made by melting first a copper manganese alloy containing 30 per cent. of copper, and adding to it the nickel and thereafter the remainder of the manganese and the antimony, tin, and lead. A deoxidant such as bronze or borax may be added during the melting.

Annealing Processes

IN the production of magnetic material having a high initial permeability and a low retentivity carbon is added to a melt comprising one or more ferromagnetic elements and annealing is then effected in presence of an agent adapted to reduce the carbon in excess of the amount required to combine with the oxygen in the material. Thus an alloy containing 45 per cent. of nickel, 30 per cent. of iron, and 25 per cent. of cobalt may be melted with 0.1–0.5 per cent. of free carbon and the product cast, rolled and passed through a wire-drawing machine, with annealing in hydrogen at 900–1,250° C. between the passes. See Specification 346,788, dated January 17, 1930, by Standard Telephones and Cables, Ltd., G. Lawton, and W. H. Dearden, of London.

Detinning Scrap

THE use as a detinning agent of a solution obtainable by dissolving spongy lead in an excess of caustic alkali solution with the aid of air or oxygen is described in Specification 346,674, dated January 14, 1930, by J. W. Hinchley, of London.



346,674

Spongy lead is precipitated and the tin goes into solution. The tin plate is fed from a shoot 22 into the alkaline liquid in a trough 60 in which rotates an outer drum 61 carrying an inner perforated drum 62 with vanes 63 which direct the scrap into a collecting trough 64. The spongy lead falls through the perforations and is discharged from an annular lip 61a. The alkaline solution is continuously circulated through filter presses.

Extracting Aluminium, etc.

IN the manufacture of aluminium from bauxite, titanium from rutile, or zirconium from monazite sand, the materials are fused by a high frequency magnetic field in a non-conducting container after a preliminary treatment to render them conductive. Such treatment may comprise preheating by other than electric means or the addition of conducting substances, such as carbon or metal powder, which volatilise or burn at high temperatures leaving no residue. See Specification 345,291, dated December 11, 1929, by Thermal Syndicate, Ltd., Wallsend-on-Tyne, a communication from Deutsch-Englische Quarzschmelze Ges., of Berlin.

Extracting Metals

IN a process for extracting metals from sulphide ores described in Specification 345,351, dated October 23, 1929, by C. Goetz, of Berlin, the ore is heated with hydrocarbon material, air being excluded, and the residue is concentrated on percussion tables, by gravitational or magnetic treatment, or by leaching. The heating may be effected in presence of a catalyst such as sulphate of iron, gypsum, or hæmatite, with

or without lime, and the hydrocarbon material used may be coal, mineral or other oils, or a gas, e.g., acetylene, water-gas or coal-gas. The gas recovered is utilised to heat the furnace. In an example, granular copper pyrites (100 parts) is heated at 600–700° C. with coal (10 parts) and sulphate of iron (1 part) to obtain after concentration grains of metallic iron and large granules of copper.

Hardening Iron and Steel

IRON and steel are hardened by treatment in a molten liquor comprising a mixture of 50 parts of calcium chloride, 50 parts of potassium chloride, and a cementation and nitrogenisation medium consisting of 4 parts of finely divided carbonaceous substance, 8 parts of alkali carbonate, and 1.5 parts of nitrogen salts such as sodium or potassium cyanide or potassium ferrocyanide. A heat protecting coating may be produced on the surface of the bath by addition of powdered calcium cyanamide. See Specification 345,677, International Convention date December 29, 1928, by A. Stähler, of Hagen, Westphalia, Germany.

Nitrogenising Steel Alloys

ARTICLES made of alloy steel are nitrogenised at 450–540° C. for 5–40 hours, and thereafter at 590–660° C. for 5–40 hours. Suitable alloys may contain up to 5 per cent. of aluminium, and up to 5 per cent. of chromium or molybdenum, or both. They may also contain up to 5 per cent. of nickel, up to 2 per cent. of manganese, up to 1 per cent. of vanadium, up to 1 per cent. of titanium, and up to 1 per cent. of zirconium. See Specification 341,912, dated October 18, 1929, by R. Sergeson, of Massillon, Ohio, U.S.A.

The case-hardening of alloy steels by nitrogenisation is facilitated by the application of a preliminary coating of a metal such as copper, silver, platinum, cobalt, or molybdenum, or a metalloid such as arsenic, or both a metal and a metalloid. The coating may be applied by electrolytic or other chemical deposition, by spraying, or by heating the steel in the vapour of the metal or metalloid. A sprayed coating should have a thickness of the order of 0.0015 inch, whereas for a coating otherwise applied a thickness of the order of 0.00001 inch is sufficient. For the case-hardening the coated steel is subjected at 450–650° C. for about 90 hours to the action of nitrogen, ammonia, or other substance liberating nitrogen. The ammonia used should be dry and its dissociation should be restricted to 20–40 per cent. The process is particularly applicable to steels containing 1–20 per cent. of chromium, and to high manganese steels. See Specification 345,659, dated December 19, 1929, by H. Sutton, A. J. Sidery, and B. Evans, of Royal Aircraft Establishment, Farnborough, Hants.

Pickling Metals

ACROLEIN derivatives of the general formula $R^1-CH=CH-CR-CHO$, in which R and R^1 are hydrocarbon radicles, are used as inhibitors in cleaning or pickling metals according to Specification 346,162, dated April 4, 1930, by Imperial Chemical Industries, Ltd., of London. α -Ethyl- β -propylacrolein is preferably employed, and may be mixed with other inhibitors or with foam producers such as sulphite cellulose waste, saponin, or molasses. A solid inhibitor preparation may be made by absorbing the acrolein derivative in nitre cake (sodium bisulphate) with or without an addition of solid sulphite cellulose waste.

Treatment of Ores

A TREATMENT particularly applicable to manganese silicate ores and to nickel and cobalt ores as a preliminary to the extraction of the metal values is described in Specification 344,492, International Convention date December 28, 1928, by Meyer Mineral Separation Co., of Pittsburg, U.S.A. The finely divided ore is damped to an extent which does not interfere with the free passage of gas between the particles and is then treated with a gas while being agitated. The damped condition is maintained throughout the treatment by the supply of further liquid, and for this purpose the liquid and gas may be supplied alternately. In examples (1) a sulphide ore containing gold, silver, lead, and zinc, is either roasted or is treated with sulphuric acid to remove part of the zinc, and thereafter is damped and treated with a mixture of air and sulphur dioxide for the formation of zinc sulphate, and (2) a tailing containing copper and cobalt is treated with water, air, and sulphur dioxide, or with air and chlorine.

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NOTICE.—Communications relating to editorial matter for our *Monthly Metallurgical Section* should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

Surface Treatment of Aluminium and Its Alloys (II)

Physico-Chemical and Electro-Chemical Methods

This article, continued from "The Chemical Age," *Monthly Metallurgical Section*, July 4, deals with processes in which chemicals are used for the production of protective surface films.

Plating and Oxidising Processes

THE methods of non-electrolytic plating of other metals on aluminium depend again on the use of special baths, but they are few in number. A copper coating may be produced by treating with boiling copper nitrate for a short time, washing and treating with a boiling bath of concentrated copper sulphate for 15–30 minutes. The articles must be transferred rapidly between the different baths and not exposed for any length of time to the atmosphere. Immersion in a mixed solution of copper sulphate and acetate also produces a copper coating. (U.S.S.R. Patent 39,783.) For a lead coating the aluminium plates are coated with lead chlorides and urea. When this mixture is heated on the aluminium the lead forms a firmly adherent coating in which any aluminium which penetrates the coating is rendered passive and not readily oxidisable. (U.S. Patent 1,362,739.)

More recently a patent has been applied for (British Patent Appl. 20,802/30) to cover a process of producing an oxide film on aluminium by immersion in an aqueous solution of an alkali together with a catalyst, with or without ammonia. A suitable bath is given as 2–6 per cent. NaOH, 1.5–3 per cent. ammonia and 2.5–12 per cent. of a catalyst, preferably sodium silicate. A number of special processes have also been brought out to provide for the surface protection of aluminium by producing coatings of complex nature. For instance, the metal surface may be treated with a lac or varnish with a synthetic resin base and containing free phenol when the phenol attacks the metal and produces a protective layer of crust. (U.S. Patent 1,754,481.) Again, the aluminium is immersed in a boiling bath containing a vanadate, molybdate, tungstate, titanate, uranate, niobate, manganate, etc., or a mixture of these salts, and an alkaline carbonate or the corresponding alkali hydroxide. (British Patent 342,256.) This process is patented in France under the trade name of "Protalisation." Another bath formula consists of a 1.5 per cent. solution of equal parts of potassium dichromate, caustic soda and alum, used at 90–95° C. Mild alkaline bath treatment is also used, one such bath consisting of 25 grams potassium carbonate, 25 grams sodium bicarbonate and 10 grams potassium bicarbonate in 1 litre of water, used hot.

Physico-Chemical Treatment

Some experimental work has been carried out to obtain a coating of zinc on aluminium by heating in contact with a suitable mixture, but the process has not yet passed the laboratory stage. There is, however, a process for the cementation of aluminium by copper, the copper being first deposited on the aluminium (by electrolytic or other means) and then heated in the electric furnace in a neutral atmosphere. No appreciable cementation takes place below 544° C. (the formation temperature of the eutectic Al-Cu in solid solution), and for any appreciable penetration the heating must increase to nearly the melting point of aluminium itself.

Simple heating in air produces a tin oxide film on aluminium and its alloys, and heating to 100–200° C. has been applied to zinc and cadmium coatings on aluminium to give rise to the formation of oxide films.

Anodic Oxidation by Electro-Chemical Methods

Many of the important methods of surface treatment of aluminium are dependent on electro-chemical processes. When aluminium is made the anode in a suitable electrolyte,

it becomes coated with a uniform, fine-textured, hard and adherent oxide film. The structure of the film contains fine pores, allowing the passage of the current, so that theoretically the process may be continued to produce a film of any required thickness; but according to recent work on the thickness of the film in relation to breakdown, a practical limit (in the region of 70μ) exists, above which the pores do not allow of equal current density over the whole surface, the film consequently being no longer uniform. Different explanations have been given of this formation by electrolysis of aluminium oxide. It is the chemical theory that the aluminium goes into solution in nascent hydrogen and precipitates the hydroxide; the electro-chemical theory depends on the nascent at the (aluminium) anode of oxygen, which combines with the hydrogen generated, hence giving rise to hydroxide formation; whilst the electrical theory is that the gaseous films forming are continually undergoing a breakdown effect, giving rise to temperatures sufficiently high to age and harden the hydroxide even to the extent of forming ultimately a crystalline oxide.

This anodic treatment is applicable not only to aluminium but to most of its alloys except those with a copper content of more than 5 per cent., and the metal may be either heat treated or aged or in the annealed condition. Modifications of the process depend on the use of different electrolytes. In Bengough's method, the electrolyte is a 3 per cent. aqueous chromic acid solution, and the voltage is raised from zero to 40 in the first 15 minutes, kept at 40 for 35 minutes, raised to 50 in the next five minutes, and kept at 50 for five minutes. In Stafford O'Brien's method, the electrolyte is a solution of a chloride in sulphuric acid; in Setoh and Miyata's method, the electrolyte is a dilute solution of oxalic acid. By this latter method, however, the aluminium has a tendency to become pitted if the circulation of the electrolyte is not perfect. The choice of method and the conditions of treatment are governed by the use for which the metal is required. The English processes are generally used for protective purposes, and the process of Setoh and Miyata is generally used for electric insulation.

Electro-Deposition

Aluminium presents special difficulties for electro-deposition by reason of its oxide film and its high position in the electromotive scale, which latter fact tends either to produce deposition of a non-adherent metal layer at the start of plating and prevent the deposit proper from adhering or to accelerate corrosion. The difficulty of the non-adherent film can be overcome by (a) coating the metal with a fast-adhering coating of a second metal (usually cadmium or zinc) to receive the coating proper, (b) working with a very big current, and (c) rendering the metal passive. The last method is most commonly used. There are also special methods which are applicable under the individual cases. The oxide film is dealt with by cleansing and pickling, the necessary processes frequently being more elaborate in the case of electrolytic deposition than in the case of other methods of coating. The metal surface is first degreased, then pickled and then burnished with powdered porcelain or pumice, being thoroughly rinsed with cold water between each process. It is subsequently coated with a thin first layer of the most suitable metal.

A method has been introduced (British Patent 339,339)

whereby the surface of aluminium is prepared for electro-deposition by electrolytic deposit of an amalgam. A suitable bath is 10 grams zinc chloride, 5 grams mercuric nitrate, 200 grams caustic soda, 200 grams sodamide, 40 grams stannous chloride and 10 grams caustic potash to 20 litres of water. The voltage required is 1.5; the time of the treatment, a few minutes.

In metal-plating aluminium, the baths must be well buffered, and contain a suitable addition agent such as gum arabic, glucose, β -naphthol, peptone or gelatine. In addition, they should have a definite pH value maintained. In general, sulphate baths are preferable to cyanide baths. Nickel, copper, cadmium and zinc are plated directly on to aluminium, and combinations of Cd-Cr, Ni-Cr, Ni-Cu-Ni, or brass, silver, gold, lead, cobalt, etc., with an intermediate plating of one of the former metals, usually nickel or copper. Nickel is plated on a roughened surface: copper, cadmium and zinc on a smooth surface.

Nickel as a Coating for Aluminium

Nickel has proved the most satisfactory metal for coating aluminium. In the first place, the coating adheres well, is dense and takes a good polish; in the second place, it can be used as a preliminary for other platings which have not so far been directly producible. In the standard methods of plating nickel on aluminium, the aluminium after ordinary cleansing is made passive by pickling in caustic soda and treating with cold or warm concentrated nitric acid. It is then sometimes washed with lukewarm water and dipped in 0.2 per cent. potassium cyanide solution until it has a silvery white matt surface, and again washed and placed in an etching bath. After etching the ultimate nickeling is carried out in baths of any of the following compositions:—(a) NiSO_4 120; NaSO_4 195; NH_4Cl and boric acid, 15 grams per litre; (b) NiSO_4 140; MgSO_4 75; NH_4Cl and boric acid, 15 grams per litre; (c) nickel ammonium sulphate 75; NaOCl 53; Sodium citrate 7.5; boric acid 15 grams per litre. Baths (a) and (b) work with a current density of 1.5 amps per sq. decimetre; bath (c) with 0.8 amps. and pH=6.

Of the metals plated indirectly to aluminium, chromium is of outstanding importance, and there is at present a big demand for chromed articles. Chromium can be satisfactorily plated over either nickel or cadmium. The chroming bath is a sulphate solution, a typical one being chromium sulphate 4; chromic acid 250; sodium carbonate 14; boric acid 3 grams per litre. The bath is used hot (50°C .) and a current density of 13–17 amps. per sq. decimetre at 4–5 volt is used.

Electrical Insulation

For requirements of electrical insulation aluminium oxide stands out among metallic oxides as a good heat resisting insulating material, and by suitable treatment of aluminium an effective coating of oxide of practically any thickness can be produced. The effective insulation depends on the hardening and thickness of the film; it also depends on the temperature and relative humidity, and for the latter reason an absolutely non-hygroscopic binding agent should be employed where oxidised aluminium wire is used in coils. For forming metallic contacts, aluminium is coated with copper by a special process, and for absorption and radiation of heat various methods of attaining a black plated surface give efficient results, the radiating or absorbing power being increased up to 50 per cent.

Coppering

In the process of coppering for electrical contact purposes, the plating has to be carried out with particular care to provide a non-porous homogeneous coating, which makes thorough contact with the base metal and is sufficiently thick to be cleaned by filing or emery paper for bolted contacts. The best results are obtained for electrical work by subjecting the aluminium surface to a preliminary oxidation treatment. After the anodic treatment, the metal is then pickled in a hot (90 – 96°C .) bath of caustic soda and sodium bicarbonate for 10–20 seconds, quickly rinsed and coppered in a bath with copper anodes. The electrolyte is a 10 per cent. solution of copper sulphate, and the current density should be 1 amp. per sq. decimetre. The material is left for $\frac{1}{2}$ to 1 hour, and to obtain sufficiently thick deposits is dried for 1 hour at 150 – 170°C . polished, and re-inserted in the plating bath.

The Institute of Metals

Annual Autumn Meeting at Zürich

THE Institute of Metals has issued a list of twenty-five papers to be presented at the annual autumn meeting, Zürich, September 14–15. This list is of record length, as the number of papers normally presented at a meeting of the Institute is fifteen. A gathering of over 200 engineers and metallurgists from all continents is anticipated. Following the meeting in Zürich—which will include works visits, receptions and other social functions—tours will be made to Lucerne, Thun, Biel and Sierre, in the course of which further works will be visited. A party will also proceed to Milan for the International Foundry Exhibition.

The complete list of papers to be presented is as follows:—Alkins and Cartwright, "Experiments in Wire-Drawing. Part I.—Behaviour of a Composite Rod"; Alkins, "Experiments in Wire-Drawing. Part II.—Notes on the Relation Between Reduction of Area by Cold-Drawing and Tensile Strength of H. C. Copper"; Blazey, "Brittleness in Copper"; Brownson and van Someren, "The Application of the Spectrograph to the Analysis of Non-Ferrous Metals and Alloys"; Cook and Larke, "The Physical Testing of Copper and Copper-Rich Alloys in the Form of Thin Strip"; Daniels, "The Attack on Mild Steel in Hot-Galvanising"; Dunn, "The Oxidation of Some Copper Alloys"; Francis and Thompson, "The Drawing of Non-Ferrous Wires"; Hanson and Slater, "Unsoundness in Aluminium Sand-Castings. Part I.—Pinholes: Their Causes and Prevention"; Hanson and Slater, "Unsoundness in Aluminium Sand-Castings. Part II.—The Effects of Using Metal Previously Subjected to Corrosive Conditions"; Houghton and Payne, "Transformations in the Gold-Copper Alloys." With an Appendix on "X-Ray Examination of Gold-Copper Alloys," by G. D. Preston, B.A.; Hume-Rothery, "The Macro-Etching of Aluminium-Silicon Alloys"; Jones, Pfeil, and Griffiths, "Nickel-Copper Alloys of High Elastic Limit"; Jones, "The Copper-Magnesium Alloys. Part IV.—The Equilibrium Diagram"; Kloninger, Keller, and Meuche, "Applications of the Electric Furnace for Non-Ferrous Metals, with Special Reference to the Bright Annealing Process"; Kurnakow and Ageew, "Physico-Chemical Study of the Gold-Copper Solid Solutions"; Murphy, "Constitution of the Alloys of Silver and Mercury"; Newson and Wragg, "Note on the Failure of a High-Strength Brass"; O'Neill, "Note on the Diameter Measurement of Certain Brinell Indentations in Cold-Rolled Metal"; O'Neill and Cuthbertson, "The Work-Hardening Capacity and Elongation Properties of Copper"; Smith, "The Spectrographic Assay of Some Alloys of Lead"; Smithells, Williams, and Grimwood, "Melting Nickel-Chromium Alloys in Hydrogen"; Sutton and Le Brocq, "The Protection of Magnesium Alloys against Corrosion"; Waterhouse and Willows, "The Effects of Cold-Rolling and of Heat Treatment on Some Lead Alloys"; V. Zeerleder, "Influence of Variations in Heat-Treatment and Ageing on Duralumin."

Detailed programmes of the Zürich meeting and of the functions at and following the meeting, may be obtained from the secretary, Mr. G. Shaw Scott, 36, Victoria Street, S.W.1.

Cobalt Production in Canada

Increasing Uses Anticipated

THE Dominion Bureau of Statistics, Ottawa, in a final bulletin reporting the production of cobalt in Canada in 1930, states that 694,163 lb. valued at \$1,144,007 were produced, this total comparing with 929,415 lb. valued at \$1,801,915 in 1929.

Ores from the Cobalt, South Lorrain and Gowganda areas of Northern Ontario still constitute the source of Canadian cobalt production, and the metallurgical plant of the Deloro Smelting and Refining Co. continues to treat the greater part of the tonnage shipped from these producing fields. The results of almost continuous research on cobalt during recent years are apparent in the many growing uses for this metal and its alloys in radio tubes, motor valves, abrasion resisters, steam turbine blades, cutting tools and other applications, especially where stress resistance under extreme temperature is a necessary factor. It is also interesting to observe that the cobalt ores of the Belgian Congo are now being smelted in Africa to form a cobalt-copper-iron alloy which is shipped to Belgium where it is reduced to metal.

Metallurgical Topics: Monthly Notes and Comments

From Our Own Correspondents

Large Ingots for Forgings

In a recent paper presented to the American Society for Steel Treating, H. H. Ashdown discusses the production of large ingots for forgings and outlines some experiments carried out to determine the chilling effect of the mould walls on the ingot metal. The relative merits of casting ingots big-end-up and big-end-down are considered. The big-end-up method is unquestionably the best where careful casting conditions are not observed, but equally good and sometimes better ingots are cast, big-end-down, where the casting temperature and rate of teeming are carefully controlled and sink-heads provided. Experience has shown that by pouring heavy ingots toward the outside of the mould, markedly superior results are obtained. This method is facilitated by revolving the mould at a slow speed during casting. In the case of octagon ingots the sonims are forced to the centre and are eventually brought into the sink head. This is also the case with cylindrical ingots, and in addition all corner defects are eliminated. The author also gives particulars of the results obtained with the use of air or water cooled moulds, and describes a method of double pouring. By this method two ladles are used placed one above the other. When the lower ladle is half empty, the upper one is opened up and the steel from this runs into the lower ladle at such a speed that the level of metal is kept uniform. This maintains over a long period a virtually steady ferrostatic head.

Faraday's Early Alloy Steels

A "FIND" of extreme interest has been made recently at the Royal Institution, and one, moreover, which in view of the approaching Faraday Centenary celebrations at the Royal Albert Hall, is particularly opportune. It is well known that Faraday made many extensive experiments with alloys of steel, not only with copper, nickel and chromium, but with the precious metals as well. Thus, he experimented with silver, palladium, platinum, and rhodium alloys with iron, many of the specimens being prepared for him, in Sheffield. Some of his results were described in a paper read by himself and Stodart, before the Royal Society, in 1822, and are freely commented upon in Dr. Percy's great work on the metallurgy of iron. Some of the specimens showed considerable corrosion resistance, and those containing nickel, or chromium, may perhaps be regarded as prototypes of the modern "stainless" steels. The find referred to is that of a box, bearing Faraday's name, and containing 79 specimens of these alloys. These were shown by Sir Robert Hadfield at the recent *Conversazione* of the Royal Society at Burlington House, on June 24 last. A paper descriptive of Sir Robert's investigations on these interesting alloys is under preparation, and cannot fail to shed an instructive light on the nature and properties of these early forerunners of the age of alloys which Sir Robert himself has done so much to promote.

Inclusions in Cast Iron

A NEW and puzzling form of inclusion in cast iron has recently been described by F. J. Cook (preprint 31-14, American Foundrymen's Association). It takes the form of bright glazy patches in castings, accompanied, when such castings are subjected to steam pressure, to a certain degree of porosity, causing water to sweat out through the glazed areas. Examination having established the fact that the open appearance of these patches, viewed under low power magnification, was due to small crystals of the inclusion having been torn out during the machining operations, it remained to discover their cause and, if possible, the remedy. A series of analyses established the further fact that the trouble was most intense in castings in which the silicon and the manganese were present in approximately equal amounts. Thus, in a typical instance, that of a large water-cooled diesel engine piston, the silicon was 1.25 per cent. and the manganese was 1.47 per cent., proportions which, in their ratio to one another, appeared to be within the danger zone. The ratio of sulphur to manganese was not found to be implicated; moreover, the shape and colour of the inclusions were quite different from those usually associated with manganese sulphide. Although the attempt definitely to ascertain, by analysis, the composition of the

inclusions, was not attended with satisfactory results, the conclusion was, on other grounds, arrived at that the inclusion consisted of manganese silicate, admixed with a little sulphur, and although it was not definitely proved to have arisen from abrasion of the linings of the cupola in which the metal was made, the suspicion was thought to be justified that the refractory linings of that vessel were really at fault, and cutting away the lining of the pipe leading from the cupola to the receiver, after each tap, and making a fresh lining with a carefully mixed refractory, was found to remedy the defect and produce sound castings.

Silver as a By-Product

THE numerous proposals which have been made of late to restore silver to its former position as a currency metal, although put forward chiefly for its important economic implications, would have hardly less importance in regard to the metallurgical implications of such a movement. According to the latest report from the Bureau of Statistics, Ottawa, the bulk of the silver produced last year in Quebec was derived from the smelting of Noranda blister copper. Silver is, indeed, the most valuable by-product of the treatment of lead, zinc, and copper ores, and upon the value realised from the recovery and sale of the white metal depends the profitable working of half the undertakings producing the three main base metals referred to. A paper published a little time ago by the Bureau of Mines, Washington, brings out clearly the economic—and therefore the metallurgical—relation of silver to other metals, in argentiferous ores. This paper, by C. W. Merrill, states that silver-bearing ores which produce 91 per cent. of the world's total silver, are also responsible for the production of 69 per cent. of the lead, 66 per cent. of the copper, and 46 per cent. of the zinc produced annually. Indeed, over one-half of the world's silver production may be considered by-product silver, as 57 per cent. of the total output of silver is obtained from ores mined, primarily, for the sake of some other metal. Cheap silver, therefore, however desirable from the point of view of the increased utilisation of the metal in the arts, and in industry, as exemplified at the recent exhibition at the Central Hall, Westminster, where its applications for chemical plant were abundantly illustrated, means greatly enhanced cost in the production of copper, lead and zinc, and the reduction of profit on those metals to vanishing point.

Rate of Testing Materials

THE speed of testing being, in all tensile tests on metallic materials, a factor capable of influencing very greatly the results obtained, a Committee of the American Society for the Testing of Materials has been engaged on investigating this aspect of the operation. When materials are tested through and beyond their elastic limits or their yield points the phenomena of plastic flow is affected by the rate of deformation. That rate is the fundamental factor in determining the elastic limit. In practice, and when making a test, the observation and control of the application of load to the specimen is the most convenient mode of giving effect to this principle. The committee has, therefore, drawn up a set of rules which enables the determination of the rate of increase of stress to be ascertained during a test, and have imposed limits which are to be regarded as maximum allowable values. These limits are, in the case of ferrous materials, embodied in "Tentative Methods of Tension Testing of Metallic Materials" (E8-27T) issued by the Society, but the warning is given that these limits are too wide for non-ferrous metals. Another direction in which the Society has cleared a good deal of useful ground is by drawing up a list of proposed definitions of terms used in relation to methods of testing.

Definition of Terms

AMONGST these the following may, in view of the confusion which often arises in their use, be specially recommended for careful consideration: *Elastic Limit*, the greatest stress which a material is capable of developing without a permanent deformation remaining upon complete release of the stress, and *Yield Strength*, the stress at which a material exhibits a specified limiting permanent set. For ascertaining the latter

property a series of recommendations are laid down for material which has a "sharp-kneed" stress-strain diagram, and for material the stress-strain diagram of which is, in the region of yield, a smooth curve of gradual curvature, respectively. For the former the "drop of the beam" method, or the "dividers method" (where an observer with a pair of dividers or other suitable apparatus watches for visible elongation between two section marks on the specimen and notes the load at which visible stretch occurs) is specified. In the case of smooth-curve materials the "set method" or an approximate method without stress-strain diagram, using an extensometer can be employed, and the mode of applying it and the precautions necessary are described.

A State Steel Industry for China

It is reported that the Ministry of Industries, in China, has drafted a plan for the establishment of a huge steelworks at Pukow, to be operated as a State enterprise. The annual import of iron and steel into China amounts to between 500,000 and 600,000 tons, and it is therefore evident that the production of iron and steel in large quantities at a low price is of essential importance in China's industrial development. Pukow is favourably situated in regard to communication facilities, having rail and water connections with many industrial centres. Pig iron and iron ore, moreover, can be brought down the river at very low cost for smelting at Pukow instead of being shipped to Japan, as is done at present. The Ministry of Industries estimates that it will be possible to have a plant in operation, within three years, capable of producing 500 tons of pig iron daily, and about 150,000 tons of steel per annum. Supplies of iron ore would be forthcoming from the Yangtze Valley, where there is an estimated deposit of 80,000,000 tons, or from Feng Huang Shan, where the resources are 4,000,000 tons. Estimates prepared by the Ministry place the total expenditure at 82,000,000 million dollars. A gross profit on the undertaking amounting to 8,000,000 million dollars, is anticipated. Sale of surplus coal to the extent of 600 tons daily at a profit of 3 dollars per ton should bring in a revenue of 650,000 dollars per annum, and 150,000 tons of steel sold at 160 dollars per ton (showing a profit of 40 dollars per ton) would bring in 6,000,000 dollars per annum; by-products are expected to show a profit of 1,560,000 dollars. The Ministry of Industries has already appointed a committee to make preliminary arrangements in this project, and this committee is at present awaiting the report of mining experts on the adjacent mineral resources.

Effect of Temperature on Metals and Alloys

In pursuance of its function as a sponsor for the exchange of information relating to the effect of temperature on the properties of metals, the Joint Committee of the American Society for Testing Materials and the American Society of Mechanical Engineers has organised a high-temperature symposium as an integral part of the annual meetings of the former society. The genesis of the Joint Committee was a prior symposium, held in 1924, and the papers presented have been written to show progress since that date and to give a comprehensive and balanced proportion of (1) the present and probable future demands of important industries for materials for service at high or low temperatures, and (2) the present state of knowledge of the properties of available metals and alloys and their industrial applications. The paper by C. A. Crawford and R. Worthington opens with a summary of results obtained by different investigators, in creep and long-time tension tests of nickel and nickel-copper, with a critical discussion of their value and an indication of certain lines of future research. The effect of elevated temperatures (*e.g.*, softening and strengthening annealing treatments) on mechanical properties at room temperature is dealt with in relation to nickel, nickel-copper and nickel-copper-zinc alloys, and data are presented on the high-temperature short-time tensile properties of typical samples of these materials. Chemical behaviour is dealt with in relation to the resistance of nickel and nickel-copper alloys to oxidation, steam, alternate oxidising and reducing conditions, activated hydrogen and nitrogen, sulphur, molten metals and fused compounds. The information given is culled from both published and unpublished data, and a useful bibliography is appended. A paper on the "Effect of Low Temperature on Metals and Alloys," by H. W. Russell,

summarises existing knowledge on mechanical properties with notes on more purely physical characteristics which have a bearing on metallurgical development. The author here discusses methods of testing and measurement of temperature and presents a detailed tabulation of the literature relating to tensile properties, hardness, impact and fatigue resistance on a variety of ferrous and non-ferrous materials. Tables are given to show the coefficients of thermal expansion at room temperature, at 100° C. and within low-temperature ranges, brief notes being added on the influence of low temperature on heat capacity, thermal conductivity and electrical resistivity. In a third paper dealing with the "Effect of Temperature on Some Properties of Iron-Chromium-Nickel Alloys," by N. B. Pilling and R. Worthington, there is a list of typical compositions of cast and wrought alloys now commercially available, with notes on the distinctive features of the various grades and on certain alloys which have been already standardised.

Nickel Alloy Steels

A SUMMARY of the properties and applications of nickel alloy steels is given in a brochure which has been published by the Bureau of Information on Nickel which is maintained by the Mond Nickel Co., of Imperial Chemical House, Millbank, London, S.W.1. In this brochure it is stated that small percentages of nickel in steel has three important effects. First, heat treatment is facilitated; secondly, mechanical properties are improved; thirdly, greater regularity of properties is obtained. In regard to heat treatment, the presence of the nickel lowers the transformation point so that the steel need not be heated to so high a temperature before quenching; it also restricts grain growth at high temperature and lessens the rate of cooling necessary for any particular quenching result. When chromium is present with the nickel, it acts in the reverse direction to the nickel. The result is that for nickel-chromium steels slightly higher quenching temperatures are necessary, but on the other hand, tempering temperatures can be somewhat raised, this often being an advantage. In quenching, the combined effect of the nickel and the chromium is rather greater than that of either of the metals alone, and thus still larger sections can be treated uniformly. Further, when sufficient nickel and chromium are present, steels may be hardened simply by cooling in air. Chromium has a hardening and strengthening effect on the steel, so that nickel-chromium steels exhibit high figures for ultimate strengths and especially for yield points. In recent years a series of steels containing molybdenum in addition to nickel and chromium has also been developed. Here the molybdenum has a very great effect on quenching facilities, and very large sections can be treated to give little or no variation in properties from outside to centre. Such nickel-chromium-molybdenum steels are not subject to "temper brittleness" as are the nickel-chromium steels.

Endurance Testing of Steel

THE Bureau of Standards in the United States is engaged in an extensive study of the properties of rail steels. One phase of the investigation is the determination of the endurance limits of this material, with the object of ascertaining, if possible, to what extent the resistance of a rail to the repeated stresses to which it is subjected in service may be related to the non-metallic inclusions or "dirt" and other unsound conditions in the steel. To determine which method of test would be the most discriminating in showing the effects of such inclusions and other unsound conditions in the steel upon the endurance properties, comparisons have been made between the rotating-beam and the axial-loading methods for the determination of endurance limits. The materials used for the comparative tests were an open-hearth iron containing 0.02 per cent. carbon; wrought iron in three stages of refinement by rolling; three plain carbon steels containing 0.45, 0.47 and 0.87 per cent. carbon, respectively, and each tested in both the annealed and in the heat-treated conditions; and four rail steels. In no case was the endurance limit determined by the axial-loading method greater than that determined by the rotating-beam method. For the 0.45 per cent. carbon and the 0.87 per cent. carbon steels in the heat-treated condition the endurance limits were practically the same by both methods of test. Indications, in general, have been that, for a non-uniform material, the endurance limit in axial loading is lower than it is when determined by the rotating-beam method.

Trade, Commerce, Finance: The Month in Review

From Our Market Correspondent

DURING the month of July there have been various factors at work affecting not only the iron and steel trade, but industry generally. In that month the quiet, but none the less real, influence of the holiday season begins to make itself felt. In most industrial districts there is what one might call an annual holiday week, when workpeople take a rest and change from their labours, and the employers give their plant and machinery a thorough overhaul and repair. The stop week varies in point of time according to local customs and circumstances, but many of these stoppages fall in July, and there is the inevitable slowing down of business activity in consequence. That is no doubt one of the chief reasons why the three summer months are generally the quietest of the whole year.

Effect of Present Financial Situation

Then there has been the more noticeable and more serious financial upheaval in Europe, which has already left its mark on international politics and economics, and may yet cause still further disturbance. With the political situation we are less directly concerned, although it is particularly unfortunate that this crisis should have arisen at a time when the trade barometer stood so low, as it has intensified the lack of confidence which has been one of the great hindrances to trade revival, and it puts back still farther the day when we may hope to take the road of recovery from the prevailing depression. Coming to the more direct effect of this international upset on the iron and steel trade, one finds that there is much apprehension in the trade as to the possible, and even probable, action which Germany's financial trouble may compel her to take. The fear is that the urgent need for money in that country may bring about a policy of forced exports, similar to what has been done in Russia.

It is stated that a fair weight of orders for steel has been passed on by the German makers to the French, Belgian and Luxembourg steel makers, and if Germany should be unable to pay for these, they would be thrown on to the export market. In addition, there is the possibility of direct dumping of steel on the European market by Germany in order to raise funds, and that would mean a break-up of continental prices, with an immediate repercussion in this country. As usual, Great Britain would stand to suffer most. France, whose selfish, shortsighted policy has been largely responsible for the trouble, would suffer less than any other country, and there would no doubt be rejoicing there if we were to be the chief losers. Germany has been given another three months' grace, but whatever the ultimate outcome may be, it is certain that we shall have no trade revival during that period, as there is too much tension in the political atmosphere to permit of any return of confidence to industry just now. If at the end of the three months a better and more lasting settlement is not made, the outlook will be pretty hopeless.

It looks, therefore, as though we shall have to face a further period, short we hope, but possibly long, of bad trade, low prices and falling outputs. The long drawn out experience we have had of this condition in the iron and steel trade is enough to bring despair to the stoutest hearted. Wherever business men congregate the same story is told and the same remark is heard: "It surely cannot go on like this much longer." Yet it goes on. The most favourably situated undertakings will have a hard struggle to get through this year without a loss, while to some it will mean final disaster. Yet another of the old-established works has recently had to fall out. The Brymbo Steel Co. is now in the hands of the Receiver, and it is doubtful whether it will be able to survive.

The Recent Meeting of Steel Associations

The third influence, in the past month, on the steel trade was the meeting of the steel associations held on July 10. That meeting had been awaited with great interest, and speculation was rife as to the outcome of it. It will be remembered

that in January last there was a strong demand from some sections of the industry for a reduction in controlled prices, and this was met by giving an increase of 5s. per ton in the rebate to those consumers who confined their purchases of steel to the associated makers, a condition of the increase being that no further change in prices should be made for six months. That period has elapsed, and in view of the serious depression, it was generally expected that the July meeting would decide to reduce prices. That expectation was strengthened by the demand put forward by the Shipbuilders Federation, who stated that they would be compelled to buy foreign steel unless the controlled prices were reduced. Nevertheless, as is now known, the steel makers decided to make no change, and they are to be commended for the wisdom of their decision. The shipbuilders had no actual business to offer, nor could they point to any which they were likely to secure by having lower prices for steel. On the other hand, there was no demand from the general body of consumers for a reduction, and it would therefore have been a serious loss to the steel makers to reduce prices all round at the request of one particular body when there would have been no compensation in the shape of increased outputs.

It needs to be borne in mind that British steel prices for plates and sections, after allowing the rebate, are not economically too high, but that other prices in comparison are too low, and the need is for some of these to be raised to a level more commensurate with the cost of production, rather than depress still further the reasonable level of the controlled prices. The decision holds good for at least three months, as there will not be another meeting of the joint associations until October. The prices will then again come under review. No doubt in the meantime the request of the shipbuilders will be given special consideration to see if some way of helping them can be found.

Conditions on the Continent

Prior to the meeting there was a tendency to hold off business in the hope of lower prices, and it is reported that some merchants even went to the length of selling at less than the prices then in force, being certain of a reduction. If that was done there need be no sympathy wasted on those who were caught short. After the result of the meeting was known there was a certain amount of forward buying, but this soon subsided, owing to the political situation, and it is probable that the industry will do nothing more than mark time until it is seen how the situation develops. The iron and steel trade abroad is not showing any improvement. In Germany, as might be expected, the depression continues, and is likely to become more acute. Outputs are down and the number of men employed in the industry seems to be steadily decreasing. Belgium is also feeling the effects of the political situation and business there is not bright. France's home trade is not materially affected, but her export trade is likely to suffer. In the United States, operations in the steel industry are now at about 30 per cent. of productive capacity, which is a rather smaller ratio than we are experiencing.

There is nothing of interest to report about actual market conditions. The pig iron trade is extremely quiet. Prices are unchanged, but orders are very scarce. There is a moderate business passing in semi-finished steel. The price for soft billets, however, is a distinctly unremunerative one, like the price of small steel bars and thin sheets. In each case, the quotations made for what business is obtainable indicate the desperate position in which the works are placed for want of orders. It would be nothing less than disastrous if these prices were to be established in the event of the adoption of safeguarding, and it is a great pity that the makers cannot devise some scheme to avoid this suicidal competition amongst themselves.

The outputs in June were down as compared with May. There were four blast furnaces less in blast, the pig iron output being 323,800 tons, compared with 346,500 tons in May. Steel production amounted to 428,900, compared with 435,100 tons in May.

Some Inventions of the Month**By Our Patents Correspondent***Abstracts of other Patents of metallurgical interest will be found in our Patent Literature published weekly in THE CHEMICAL AGE.***Alloys**

An alloy suitable for addition as a hardening ingredient to copper or its alloys consists of 5-70 per cent. of iron, 20-95 per cent. of copper, and 10-65 per cent. of silicon, with or without 0.1-10 per cent. of phosphorus. The addition is of such an amount that the hardened product contains up to 5 per cent. of silicon and up to 11 per cent. of iron, and the phosphorus content may be up to 1 per cent. See Specification 347,098, dated December 12, 1929, by U. De Berker, P.-M.-G. Metal Trust, Ltd., W. Machin, and W. B. O'B. Goudielock, of London.

MAGNETIC alloys described in Specification 347,305, dated January 30, 1930, by Callender's Cable and Construction Co., Ltd., of London, and S. Beckinsale, of Belvedere, Kent, comprise 65-80 per cent. of nickel, 15-33 per cent. of iron, 0.05-10 per cent. of cobalt, 0.05-15 per cent. of silicon, and 0.05-10 per cent. of molybdenum. An alloy suitable for drawing into wire contains 75 per cent. of nickel, 23.4 per cent. of iron, 0.5 per cent. of cobalt, 0.01 per cent. of silicon, and 1 per cent. of molybdenum, and one suitable for the production of powder contains 72 per cent. of nickel, 16 per cent. of iron, 2 per cent. of cobalt, 7 per cent. of silicon, and 3 per cent. of molybdenum. They are annealed, after being worked, by heating at 875-925° C. in absence of oxygen and thereafter cooling slowly to about 625° C.

ALLOYS for making commutator bars of dynamo-electric machines, described in Specification 347,080, International Convention date August 10, 1929, by Associated Electrical Industries, Ltd., of London, a communication from G. A. Moore, of Wilkinsburg, Pennsylvania, U.S.A., contain copper and cadmium, the copper content being not less than 90 per cent. and preferably 95-97 per cent.

Annealing Silver Alloys

SILVER alloys containing 4.5-25 per cent. of manganese are heated to above 600° C. but below fusion temperature and thereafter, preferably after quenching, are allowed to age at 400-100° C. to improve their strength and hardness. The operations may be repeated with or without further consolidation by rolling or pressing. The process is applicable to commercial silver-copper alloys containing not more than 4 per cent. of copper and to alloys in which part of the manganese is replaced by not more than 5 per cent. of aluminium or not more than 8 per cent. of zinc, cadmium, thallium, antimony, tin, arsenic or bismuth. See Specification 346,451 (Johnson), dated January 13, 1930, a communication from I.G. Farbenindustrie Akt.-Ges., of Frankfurt-on-Main, Germany.

Extracting Metals

THE treatment of metal halides with hydrogen to obtain the metals is described in Specification 346,921 (Johnson) dated February 27, 1930, a communication from I.G. Farbenindustrie Akt.-Ges., of Frankfurt-on-Main, Germany, as carried out in apparatus of which at least the heat-conveying parts are made of iron or iron alloys. The production of iron, chromium, and copper from their chlorides is specified, and the use of the chromium so obtained in the manufacture of acid-proof steels is referred to.

Iron

THE magnetic properties of iron are improved by a heat treatment *in vacuo* below its melting-point but above 400° C. and preferably between 600° C. and 900° C. according to Specification 347,304 (Johnson), dated January 30, 1930, a communication from I.G. Farbenindustrie Akt.-Ges., of Frankfurt-on-Main, Germany. The iron used is such as is obtainable from the finely divided metal by sintering and the application of pressure and/or heat, preferably in an atmosphere of hydrogen. Iron powder obtained from iron carbonyl or from iron chloride is suitable. The iron may be subjected to working in the cold by rolling or stretching prior to the heat treatment.

Purifying Magnesium

PURIFICATION of magnesium or of a magnesium alloy is effected by heating the metal in a relatively large bath of

molten flux to a temperature at which it is partially melted and thereafter agitating the bath to cause the particles of metal to coalesce. The temperature may then be raised to enable the metal to be further washed with the flux. Successive baths of progressively cleaner flux may be used for such washing. A suitable flux comprises magnesium chloride or a mixture of magnesium and sodium chlorides with or without barium chloride, or potassium chloride and calcium fluoride. See specification 346,271 (Arnold), dated December 9, 1929, a communication from Dow Chemical Co., of Midland Michigan, U.S.A.

Refining Metals

To purify metals of low melting-point from metallic impurities they are heated to a temperature which is sufficient to melt the mass but is below the melting-point of alloys of the metals present with each other as impurities, and the metal is then filtered while maintaining the main mass molten. An addition may be made of a metal capable of alloying with the impurities. The purification of lead from admixture with copper, tin, and antimony by heating to 327° C. and filtering off the solid copper-tin and copper-antimony alloys is specified. Lead and tin alloys used as bearing metals may be purified in two stages, viz., (1) heating to a temperature slightly above the melting-point of the main mass and filtering off the solid copper-tin alloy, and (2) adding aluminium to the filtrate to combine with antimony and copper and filtering off the aluminium-antimony and aluminium-copper alloys. The separated copper tin alloys may be used as bearing metals. Suitable filtration plant is described. See Specification 347,222, dated January 24, 1930, by A. Henderson, of Birmingham.

Treating Antimony Ores

To separate gold and antimony from sulphide or other ores of antimony the ore or matte is mixed as a finely crushed concentrate with a flux such as nitre cake, salt cake, an alkali metal bisulphate, magnesium sulphate, aluminium sulphate, soda or potash alum, sodium sulphate and sulphuric acid, or common salt and sulphuric acid, and the mixture is roasted with a regulated supply of air; the mass is then leached with water and the residue is treated by known methods for recovery of the gold. Thus 1 part of a concentrate containing the gold as metal or as sulphide, arsenide, telluride, selenide, etc., may be mixed with 2 parts of the flux and the mixture be charged into a furnace at 100-150° C. and roasted until all the sulphides, arsenides, and antimony are oxidised, the final and maximum temperature of the mass being not above 450-500° C. The cooled mass is agitated in cold water, and the insoluble residue is collected on a filter. To ensure a high recovery of gold by use of mercury the residue should be slimed before or during amalgamation. The water insoluble portion after removal of the amalgam may be treated for extraction of the antimony, e.g., by drying and reducing with fluxes to give a regulus. Some antimony is also recoverable from the leaching liquor by agitating it with milk of lime or magnesia and heating to effect a rapid precipitation. See Specification 347,680, dated January 27, 1930, by H. W. C. Annable and Ferro Alloy Co. of Africa (Proprietary), Ltd., of Port Elizabeth, South Africa.

Zinc

A PROCESS and plant for the extraction of zinc from its ore or by the use of a continuous electrically heated shaft furnace are described and illustrated in Specification 347,239 (Triggs) dated December 17, 1929, a communication from St. Joseph Lead Co., of New York, U.S.A. The granular free-flowing charge, which is preferably preheated, comprises a mixture of granular sintered zinc ore and granular carbonaceous material, and is heated in the furnace, by utilising its resistance to the current, to a temperature sufficient to distil off the zinc but insufficient to fuse or slag the residue. Such temperature is preferably below that at which the sulphides in the ore are distilled, e.g., about 1,100° C. The sintering of the ore may be effected in conjunction with a sulphurising process. The furnace described is constructed of superposed sections each supported independently, the uppermost section constituting the preheating chamber, a number of intermediate sections the reducing chamber, and the lowermost section the discharge chamber. Provision is made for oxidising the zinc vapour with air when zinc oxide is required.

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The Manufacture of Chemical Sheet Lead

By R. S. Russell

The following extracts are taken from a recent paper read before the Melbourne University Metallurgical Society. The author is metallurgist to Broken Hill Associated Smelters Pty., Ltd., and has an intimate knowledge of the manufacture of chemical lead intended for service in the construction of chemical plant.

THE term "chemical lead" signifies a highly purified form of lead, usually containing not less than 99.99 per cent. Pb. The consumer who desires to purchase lead of this quality will find two standard specifications to guide him—one British* and one American.† Both specify the chemical composition only. No physical properties are specified, partly because lead is so weak that it can hardly be regarded as a constructional material at all, but only as a lining, and partly because there is no clear understanding of the action of that physical property which is by far the most important, namely, fatigue limit. Furthermore, there is no simple method of measuring fatigue limit of sheet lead even if that property could be specified.

The British Standard Specification

The British specification recommends two accelerated corrosion tests, which are alleged to furnish an index of the ability of lead to resist corrosive influences, particularly those encountered in sulphuric acid plants. Experience has shown, however, that these tests are quite unreliable, in fact, that they may be positively misleading. This British specification calls for a higher standard of chemical purity than the American. However, the author considers that neither is quite correct, and suggests the following maximum limits for any one impurity:—Silver, 0.002 per cent.; bismuth, 0.005; iron, 0.003; antimony, 0.004; zinc, 0.002; copper, 0.001; nickel and cobalt together, 0.001; tin, cadmium, arsenic, traces. The lead content, obtained by difference, must be not less than 99.99 per cent.; hence, if some of the above-mentioned impurities are up to the limit, then others must be below it, so that the total of impurities may not exceed 0.01 per cent.

It is not to be suggested that the purchaser of chemical lead should check its composition, for the analysis is tedious and expensive, and requires the services of an experienced analyst. In Australia, at least, there is no need for him to do so, for the composition of the lead is so controlled during refining and so checked before shipping that there is very little chance of lead less than 99.99 per cent. pure being sent out. The fabrication of the lead ingots into pipe or sheet requires the exercise of every care to prevent contamination of the metal, but, given proper diligence on the part of the manufacturer, there is no likelihood of impurities being introduced into the lead.

Manufacturing Procedure

The method of rolling chemical sheet lead is briefly as follows:—Lead ingots, virgin from the refiner, are melted in an iron pot that has been cleaned and scraped down. The pot is usually oil-fired, and the flames pass from the pot to heat the mould. The correct temperature of casting is about 450° C. If the lead be too hot the crystals may split apart during cooling; if it be too cold, the casting will be laminated, and either cracks or laminations, whichever may be present, will survive the rolling process, and will cause weakness in the final sheet. The melt is skimmed before pouring, and skimmed again after casting. This second skimming of the lead in the mould is important. Lead at 450° C. can dissolve an appreci-

able amount of lead oxide, which separates on cooling, and which must be given every opportunity to rise to the surface. Hence if the mould be so cold as to chill the lead, or if the skimming be not done carefully, lead oxide will be entrapped, and will impair the continuity of the sheet.

The cast block measures 6 ft. square; it is 6 in. thick, and weighs 10 tons. It is lifted and rolled while hot. This hot rolling is done as soon and as quickly as possible, for the reasons that hot lead requires less power to roll it, and warm lead anneals itself (and thus relieves internal stress) even more readily than cold lead. The only danger in hot rolling is that the crystals may split apart; however, the lead would need to be near the melting point for this to happen. The product of hot rolling is a slab 1 in. thick known as "stock sheet," which is cut by a guillotine and kept for cold rolling to sheet as and when required. Sheet destined for use in, say, a sulphuric acid plant is usually $\frac{1}{8}$ in. thick, and is designated as 8-lb. lead, which signifies that 1 sq. ft. weighs 8 lb. Such sheet should be finely and uniformly crystalline.

Fatigue Limit of Chemical Lead

The fatigue limit of lead, determined on the Haigh machine, is ± 0.2 tons per sq. in., that is, 10,000,000 reversals of a stress of 0.2 tons per sq. in. will cause failure at ordinary temperature. At 100° C. the fatigue limit is about four-fifths that at ordinary temperature, and at 150° C. it is reduced to less than half.

It has become evident that, if lead be subjected to vibration, the oscillatory stresses set up by the motion of one part in relation to another will cause intercrystalline cracking. The frequency and the amplitude of vibration needed have not been worked out, but apparently these factors can vary within wide limits. The movement may be so small in amplitude that it could be detected only by special means, or it may be so slow as to escape notice. This is quite feasible when one considers the length of time for which the vibratory stresses may be operating. The idea has also been expressed that fatigue failure is due to "crystallisation," by which is meant, presumably, "grain growth," although the author has never seen a sample of failed lead in which abnormal grain size was associated with the cracking. It may happen, however, that sheet lead is covered with a film of fine lead crystals, underneath which the true grain size may be found by etching off the superficial film.

Remedy for Fatigue Failure

The remedy for fatigue failure lies in (1) elimination of vibration, (2) working at as low a temperature as possible, (3) preventing changes of temperature, and (4) supporting the lead as completely as possible. It has been the author's experience that fatigue is the predominant factor in most failures of lead, though, of course, simultaneous corrosion may accelerate the effects of fatigue. It is a general rule, however, that the purer the lead the greater will be its resistance to corrosion in chemical plant. Though the purer lead is the more susceptible to fatigue, it is better to use pure lead, and to minimise fatigue by proper design of the plant, because most elements, which could be added to lead to improve its fatigue resistance, would probably spoil its corrosion resistance entirely.

* British Engineering Standards Assoc. Specification No. 334, 1928.

† American Soc. Testing Materials, Standards 1927, Part 1, p. 527.

The Iron and Steel Industry

A Comparison of World Conditions

ACCORDING to the *Statistical Bulletin* issued by the National Federation of Iron and Steel Manufacturers, the production of pig iron in the United Kingdom during July amounted to only 317,000 tons, compared with 323,800 tons in June, and 486,100 tons in July of last year. There was a net decline in the number of furnaces blowing to the number of six, reducing the number in operation to 70, which is lower than at any time since the resumption after the stoppage of 1926. Steel production totalled 428,700 tons, or practically the same as in June. Imports of iron and steel declined by about 15,000 tons to 231,000 tons, and exports increased by 5,700 tons to 168,300 tons, so that the excess of imports over exports, which in June amounted to 83,700 tons, declined to 62,700 tons. The items chiefly responsible for the decline in imports were pig iron, which declined by 8,800 tons to 19,800 tons, and sheet and tinplate bars, which declined by 18,100 tons to 63,900 tons. Imports of billets, blooms and slabs, of steel bars and of plates and sheets showed an increase. The chief feature in the export returns was the welcome increase in the export of tinplates and sheets from 25,700 tons in June to 37,800 tons in July.

Germany

The abnormal economic and financial conditions obtaining in Germany have caused almost complete stagnation on the domestic steel market. Some merchants are stated to have been cutting prices in an endeavour to unload their stocks. On the export market rather more activity has been noted, but the volume of business continues to be very disappointing, and it is feared that the keen competition between Continental steel manufacturers will result in weaker prices unless there is a pronounced improvement in the demand from the overseas markets, of which there appears to be no sign at the present time.

France

In France there is little change to report in conditions in the iron and steel industry; the recent curtailment of output has resulted in some increase in unemployment and short time. New orders are scarce and cover only immediate needs. At the present time consumers have no inducement to place long period contracts in view of the fact that producers are able, in the majority of cases, to give immediate delivery out of accumulated stocks. The Saulnes Works in France have given notice of withdrawal from the Franco-Belgian-Luxemburg syndicate which controls the sale of foundry pig iron on the Belgian market. Any member had the option of announcing its withdrawal from this syndicate up to July 31; it was provided, however, that withdrawal should not take effect until October 1, when all members of the syndicate would be free to deal on the market on their own account. It does not follow, however, that this will happen, for it is understood that negotiations are proceeding between the Saulnes works and the other members of the syndicate with a view to reaching a mutually satisfactory arrangement. This may possibly involve the granting of a higher quota to the Saulnes concern. It remains to be seen whether these discussions will be successful, but it is pointed out that the reconstitution of the French foundry pig syndicate would be an important favourable factor.

United States

According to the latest cabled information received from the United States, the demand for steel has been more encouraging and prices have remained steady. Business in structural material and pipe line has been more active than for several months past. There has also been more interest shown in the market for railway equipment. The net earnings of the United States Steel Corporation in the second quarter of 1931 amounted to £1,520,000 (including £1,470,000 special income which was derived from the sale of the property of a small Public Utility Company), compared with £1,390,000 in the first quarter and £6,600,000 in the second quarter of 1930. The Bethlehem Steel Corporation has acquired the properties and business of the Kalman Steel Co., subject to the approval of the stockholders of the latter. The Kalman Steel Co. is a large fabricator and distributor of reinforcing steel, with warehouses in various cities in the East and Middle West.

Recovery of Metals from Secondary Sources

United States Statistics for 1930

THE value of certain non-ferrous metals recovered from secondary sources during 1930 is reported to the United States Bureau of Mines as \$193,255,100, which is \$137,773,800 less than in 1929, and the total quantity decreased 24 per cent. This decrease in value was partly due to the lower yearly average prices of copper, lead, zinc, tin, and antimony in 1930. The smelters that treat mainly primary material produced 26,809 tons less secondary copper in 1930 than in 1929. There was a decrease of 26 per cent. in secondary lead reported by regular smelters that treat mainly ore and bullion, and there was also a large decrease in the output of lead in alloys reported by smelters that treat scrap and drosses. Zinc recovered by re-distillation decreased about 12,500 tons, and that recovered by re-melting and sweating decreased about 3,600 tons. There were also decreases in the quantities of zinc dust, zinc chloride, and zinc sulphate made from zinc residues and zinc dross. The de-tinning plants treated about 13,400 tons less clean tinplate clippings in 1930 than in 1929, and the yield per ton increased considerably. About 69 per cent. of the recoveries at de-tinning plants was as tin content in tin tetrachloride, tin oxide, and other chemical compounds, compared with 76 per cent. in 1929. The constantly declining average monthly prices of nearly all metals made the year generally unprofitable for secondary metal dealers and smelters.

The Iron and Steel Institute

Swansea Meeting

THE autumn meeting of the Iron and Steel Institute will be held at the Royal Metal Exchange, Swansea, September 29 to October 2, when the following papers will be submitted:—"The Equilibrium of Certain Non-Metallic Systems," Part I, by J. H. Andrew, W. R. Maddocks and D. Howat, Part II, by J. H. Andrew, W. R. Maddocks and E. A. Fowler; F. Adcock: "Alloys of Iron Research, Part X.—The Chromium-Iron Constitutional Diagram"; C. O. Bannister and W. D. Jones: "The Diffusion of Tin into Iron with Special Reference to the Formation of Columnar Crystals"; N. T. Belaiew: "The Structure of Nodular Troostite"; G. Burns: "The Effect of Molybdenum on Medium-Carbon Steels Containing 1 to 2.5 per cent. of Manganese"; O. Cromberg: "Production Economy in Iron and Steel Works, Part II.—Costs of Production"; W. H. Cunningham and J. S. Ashbury: "The Surface Hardening by Nitrogen of Special Aluminium-Chromium-Molybdenum Steels on a Production Basis"; C. A. Edwards, F.R.S., and A. Preece: "The Constitution of the Iron-Tin Alloys"; G. A. Hankins and M. L. Becker: "The Effect of Surface Conditions Produced by Heat Treatment on the Fatigue Resistance of Spring Steels"; R. Harrison: "The Influence of Silicon on Nickel Steel"; J. C. Jones: "Mottled Tinplates"; B. Matuschka: "The Solidification and Crystallisation of Steel Ingots: the Influence of the Casting Temperature and the Undercooling Capacity of the Steel"; F. C. Thompson and R. Willows: "A Critical Study of the Origin of the Banded Structure of a Hot-Worked Hypoeutectoid Steel."

Termination of Aluminium Cartel Probable

ACCORDING to information from the Continent, the International Aluminium Cartel, which comprises the most important producers in England, Germany, France, Switzerland, Austria and Norway, is expected to come to an end at the close of this year, when the agreement expires. The chief reason is attributed to the expansion of American aluminium interests. The Aluminium Co. of America is the sole producer in the United States, and Aluminium Ltd., formed by the segregation of the foreign properties of the Aluminium Co., is the only other aluminium producer in America. Of the ten nations of the world which produce aluminium in commercial quantities, the United States is the largest producer, and last year Canada was the second largest producer, giving North American producers approximately 50 per cent. of the world output. Last year European production was as follows: Germany, 30,900 tons; France, 29,000; Switzerland, 22,400; Norway, 20,500; Great Britain, 19,400; Italy, 8,000; Austria, 3,500 tons, and Spain 1,000 tons. The price of aluminium ingots and bars in this country has remained at £85 per ton over a long period.

Metallurgical Topics: Monthly Notes and Comments

From Our Own Correspondents

The Faraday Centenary

THE Faraday Centenary celebrations have now commenced, and will continue all the month. The event of most importance to metallurgists is the Exhibition which is to be held at the end of the month at the Royal Albert Hall. Apart from the exhibits of electrical plant, equipment and machinery, which will occupy most of the space, an apportionment has been made to the allied branches of electro-chemistry and electro-plating developments, both of which owe their origin and modern progress to the laws of electrolysis which Faraday was the first to enunciate. The sections devoted to modern aspects of electro-chemistry—so far as they relate to electro-metallurgy—comprise, of course, electro refining, but apart from the furnaces, etc., exemplifying the applications of electricity to the melting and the heat treatment of metals, space has been devoted to a highly representative display illustrative of the electrolytic recovery and refining of metals. In the organisation of this exhibit, the British Non-Ferrous Metals Research Association has taken a leading part, and its Director, Dr. R. S. Hutton, has succeeded in inducing most of the leading mining and metallurgical firms engaged in the production of non-ferrous metals, at home or in the Dominions, to show their products and processes in this Section.

Modern Developments in Electro-Plating

ADJOINING this section will be one devoted to another branch of electro-metallurgy which similarly owes much to Faraday. This is the electro-deposition and the electro-plating section. There, too, Dr. Hutton, who is President of the Electroplaters' and Depositors' Technical Society, which has organised this section of the Exhibition, has given invaluable aid, and a highly representative display will be made, to which a number of the leading firms in the country have contributed. Amongst the exhibits will be small scale models of nickel and chromium plating plants, together with one automatic plating plant, which exemplify present-day practice in the mass production of plated articles. Another exhibit of extreme interest will be that of the Research Department, Woolwich, representative of investigations into the nature of research into their control and improvement. To catalogue all the specimens which will be shown would be exhaustive, but mention should be made of those showing the decorative aspects of electro-deposition, as distinct from other exhibits illustrative rather of the protective nature of metallic coatings deposited by electrolytic methods, and a number of specimens of plating with platinum group metals, which has only recently become a commercial proposition consequent on the fall of prices of these metals. The specimens shown will illustrate the most recent advances in the plating of palladium and rhodium, the latter being not only exceedingly pleasing in appearance, but practically untarnishable. Finally, reference should be made to an exhibit exemplifying the building-up of undersized or worn parts by electrolysis, and the application of that method to the coating of machine components and structural parts with a metal harder than the original base, and hence conducting to better wear and longer life of the parts so treated. The Royal Mint, the Victoria and Albert Museum and the London School of Printing are also contributing displays of artistic and other kinds of electrotyping.

Artificial Gold

THERE has, during the last few weeks, been a revival of interest in "artificial gold"—not, it is to be hoped, a distressing sign of the times, emphasising the lack of the genuine metal, but rather as a subject *pour vivre*. The *Times* published a note to the effect that successful experiments had been made in Sheffield in the casting of an aluminium-copper alloy that had the appearance of gold and that could be varied somewhat to imitate the real gold alloys of different caratage. This aluminium bronze, to give the alloy its real name, has been known for half a century or so, and formed the subject of lectures at the Royal School of Mines in the '90's. Samples prepared in Dr. Percy's laboratory in 1865, and now in the Science Museum at South Kensington, still retain their good colour. The useful series of aluminium bronzes contains up

to 10 per cent, or so of aluminium, with or without small amounts of iron, nickel, manganese, etc. They have for some time been used for ornamental articles and cheap jewellery, on account of their close resemblance to 22 carat gold. What is of far greater importance is that alloys of this type are finding considerable use commercially in all forms, sand and chill castings, stampings, pressings, forgings, rods, tubes and sections and sheets, and have a high strength-weight ratio.

White and "Fancy" Golds

WHILE artificial gold is used for some kinds of cheap jewellery it must not be confused with "white gold" the legitimate use of which is practically confined to the goldsmith's trade. It has long been known among goldsmiths and jewellers that small proportions of other metals profoundly alter the colour of gold. Of "white golds" there are many, all being entitled to the hall-mark denoting their proper caratage. Thus there is an 18 ct. white gold, containing 16.5 per cent. of nickel, 5 per cent. of zinc and 3.5 per cent. of copper; a 12 ct. alloy containing 20 per cent. nickel, 20 per cent. copper and 10 per cent. zinc, with a minute amount of manganese. In other 18 ct. varieties the nickel content is slightly increased at the expense of the copper. A truly noble white gold—21.6 ct.—contains 90 per cent. gold and 10 per cent. palladium. Coloured golds of various hues are also used in the making of high-class jewellery, particularly for floral designs. By selecting the proper alloy a whole range of colours becomes available. Even a puce colour is said to be obtainable with a 20 ct. alloy containing one-sixth of silver. The use of iron, platinum or cadmium as the alloying metal gives various shades of grey. Incidentally, aluminium finds a use here as in artificial gold. Alloyed with gold to make a 22 ct. alloy the resulting melt is of a pale violet colour, hard and brittle but quite useful for casting purposes. This alloy is a metallurgical curiosity, since the union of aluminium with gold—both very malleable in the pure state—results in an excessively non-malleable alloy.

An Ideal Metallurgical Paper

THE secretaries of most learned societies have learned by hard experience to wean the authors of papers from saying what they have to say too greatly by the aid of illustrations. In all too many metallurgical papers there is still, however, an exasperating tendency to use too many illustrations of the photo-microscopical sort, where, very often, a letterpress description of the appearances noted would suffice. The same objection arises when too many diagrams, particularly of graphs derived from numerical data, are used, especially when the data used for plotting them are also embodied in tables, which are expensive to set. Both are very rarely necessary. The ideal paper, in these respects and in the manner of its arrangement and presentation, is hard to seek. Nevertheless, a paper closely corresponding with the ideal was presented at the Chicago meeting of the American Foundrymen's Association. This is not a learned, in the sense of a "highbrow," society; it exists for much the same purposes as our own Institute of British Foundrymen. The paper referred to was by Roy M. Allen, and was entitled "The Microscope as a Practical Aid in the Cast Iron Foundry." The author treats his subject in an elementary fashion; the paper is one which can be read and understood by the journeyman and the apprentice. It is, moreover, magnificently illustrated, and no one will grudge the expense to which the Association will have been put in the matter of reproducing the photomicrographs Mr. Allen has introduced to elucidate his subject.

Constituents of Cast Iron

THE paper itself, compressed into 84 pages, of which many are taken up with page-sized photomicrographs, contains more, and better expressed information on the subject of the constitution of cast iron, and the factors that control that constitution, than many a bulky text book devoted to the subject, and gives, in addition, very useful hints to the foundryman, turned microscopist, as to the use of his instrument, and modes of polishing and etching his specimens. The paper is not meant

to be encyclopædic; it would have defeated its purpose had it been so, besides failing to appeal to the kind of reader to whom it is addressed. It is, however, immensely suggestive, and constitutes an admirable introduction to the subject with which it deals. It emphasises the importance of the rôle played by silicon, in graphitisation, and indicates the mode of controlling that still abstruse phenomenon. It gives accurate and simple descriptions of all the leading constituents of cast iron and explains what they are and how they are formed. It also discusses the influence of the other customary elements, sulphur, manganese and phosphorus, present in cast iron, as well as that of added elements such as chromium and nickel, always from the point of view of elucidating the nature of cast iron, its behaviour and properties. The photomicrographs are excellent. Few readers of the paper, coming to the subject with fresh and perhaps unformed minds, will be content to leave their knowledge of it where the author has. On the other hand, it may safely be prophesied that what further progress they make will be due to the stimulating interest attaching to Mr. Allen's paper which is of a kind which may well be taken as an example to prospective authors of papers generally.

New Outlets for Mercury Desired

THE reduction of the price of mercury at the beginning of July from the level of £21 15s. per 76 lb. flask f.o.b. Spanish or Italian port, which had been maintained by the cartel for some three years, to the present figure of £16 15s., was a move for which consumers had been agitating for a considerable time. How far lower prices will stimulate consumption is a very doubtful point, according to a special correspondent writing in the *Manchester Guardian Commercial*. It has been suggested that the cartel have their eye on the possibilities offered by the mercury boiler, which has reached a high stage of technical perfection in the United States. Probably also the growth of the American production, which has always responded to the stimulant of high prices, was causing them some concern. Thus the American consumption sank from 38,500 flasks in 1929 to 25,200 flasks in 1930, and the imports from Europe, which previously supplied a very large part of the consumption, were virtually negligible. Spain holds, in effect, the control of the world's mercury position. The Italian output is of approximately the same amount, but the Spanish ore is much the richer of the two, averaging 5 to 8 per cent. of mercury against somewhat under 1 per cent. in the case of most of the Italian ore. Negotiations for the formation of the Italo-Spanish cartel (*Mercurio Europeo*) were completed in July, 1928, and the cartel came into force on October 1, 1928. It always claimed, by way of justifying its policy, that it had stabilised prices which previously had always been subject to violent fluctuations due to speculation. While this is undoubtedly true, it has been suggested that it was the Italian influence which led to the price being maintained at a level which must have shown a very lucrative margin over the cost of production even before the modernisation of the Spanish mines was taken in hand. Rumours of the dissolution of the cartel, which were current about the middle of 1930, were denied; but there seems little doubt that there was a certain restiveness on the part of the Spaniards, who did not like to see the accumulation of heavy stocks taking place, and that with the change-over to the Republican regime this feeling became accentuated, and the situation was such that it was found expedient to decide on a stoppage of operations at Almaden from March, 1930, for about eight months. While accurate details of the Spanish output in 1930 are not yet available, it is believed that the figure will be about 58,000 flasks (of 76 lb.) against 71,750 flasks in the previous year. One of the characteristics of the mercury trade is that there are no outstandingly large uses for the metal. *Mercurio Europeo* has made various efforts to increase the consumption, as by offering a prize of £5,000 for any discovery which will lead to the use of 5,000 additional flasks by 1935. Since, however, the largest outlet in the United States—the manufacture of drugs and chemicals—only consumed 8,500 flasks in 1929, it does not look as if the prize could be easily won.

Special Refractories for Pure Metals

IN the determination of the physical properties of a pure metal it is usually necessary to melt the metal in order to secure suitable specimens. It is therefore highly desirable

that no impurities be introduced into the metal from the crucibles in which it is melted, and crucibles of special oxides or of unusually purer refractories, which are not commercially obtainable, are frequently required. The refractories ordinarily employed in the commercial melting of metals are not suitable for the melting of very pure metals at high temperatures. In the oxides of zirconium, magnesium, thorium, and beryllium, however, there are refractories available which have been used for the melting of metals at temperatures up to 2,200° C. without contamination of the metal by the refractory. A paper in the June number of the *Bureau of Standards Journal of Research* describes methods for the preparation from these refractory materials of various shapes that are used for the laboratory melting of very pure metals. The refractory oxide mixed with a suitable bonding material is tamped, pressed, or otherwise formed to the required shape and then fired at a temperature which should be somewhat higher than that at which it is subsequently to be used, in order to minimise further sintering and shrinkage in service. A sample of pure platinum contained in such a crucible of fused thorium oxide was melted and allowed to solidify more than 150 times in the course of a determination of its melting point, without loss of any of the metal by leakage through the crucible, and the purity of the platinum was practically unchanged at the completion of these tests.

Sulphur-Resistant Nickel Alloys

IN a recent contribution to the literature of heat-resisting alloys (*Zeitschrift für Metallkunde*, May, 1931), H. Gruber discusses the nature of the attack of sulphur compounds at high temperatures on iron, nickel, chromium, cobalt, silver, molybdenum and nickel-chromium alloys of simple and complex type. Detailed results are given of tests in which these materials were exposed, in comparison with materials of similar type but containing additions of aluminium, to the action of sulphur-bearing atmospheres, at temperatures of 700°, 800°, 900°, and 1000° C. Addition of aluminium, in almost all cases, very markedly lessens susceptibility to corrosion; tests of alloys containing 5, 10 and 15 per cent. aluminium indicate that in the majority of cases 10 per cent. is a specially favourable content. The brittleness resulting from a high aluminium content to some degree restricts the number of these alloys which are of practical utility, but the compositions tested reveal the possibility of a highly useful range of casting materials; an alloy containing nickel 51, chromium 15, iron 20, manganese 4, aluminium 10 per cent. appears to offer a specially desirable combination of properties and to be suitable for very varied application.

New Uses for Thallium

THALLIUM is one of the rarer metals that has come into commercial use within recent years. Its comparative scarcity, combined with a resemblance to the common metal lead, have retarded development, and although production is still measured in pounds rather than in tons, the metal is now finding application in various fields. In its physical and chemical properties it is very similar to lead. The metal is of a white colour with a bluish-grey tinge, somewhat paler than lead. It has a bright metallic lustre when freshly cut, but dulls quickly when exposed to air. The supply was formerly obtained from flue dusts that accumulated in sulphuric acid plants where pyrite is used, the thallium content of these flue dusts being usually about 0.5 per cent. At present, however, most of the domestic supply in the United States is a by-product of the purification of cadmium, itself a by-product of the smelting of other metals. Alloys of lead and thallium are somewhat unusual in that they have higher melting points than either of the component metals; they are used in rather small quantities in special types of electrical fuses. An alloy containing 10 per cent. thallium, 20 per cent. tin, and 70 per cent. lead is resistant to the corrosive action of mixtures of sulphuric, nitric, and hydrochloric acids. This alloy has been recommended for use as an anode for the electrolytic deposition of copper, since its corrosion is less than one-fifth that of lead alone. Experiments also indicate that the addition of thallium to lead-base alloys markedly improves their resistance to deformation. Further details are given in *Information Circular* 6453, recently issued by the United States Bureau of Mines.

Trade, Commerce, Finance: The Month in Review

From Our Market Correspondent

AUGUST is usually an unsatisfactory month for the iron and steel trade and for that reason one is not sorry to see the end of it. In times of good trade there is a noticeable slackness at that period of the year, which seems to be the culmination of the holiday season; but in times of depression such as we are experiencing, the slackness is intensified, as there is so little in prospect when the stoppages are over. It is only the urgent requirements that cannot be postponed which are given out, and they are kept down to the absolute minimum. The necessity for economy is not only national but individual, and nowhere more than in the steel works has it been incumbent to cut down every item of unwarranted expense; indeed, many legitimate items have had to be curtailed in the attempt to make ends meet. It is obvious, therefore, that there can be no encouraging movement to report in the iron and steel trade on this occasion. Prices have been maintained, probably because most makers are beginning to realise that it is utter folly to go on cutting for the small amount of business available; and surely some of the steel prices are now at a level which can only be described as ridiculous.

Efforts to Maintain Steel Prices

The pig iron makers have been gradually reducing their output to keep pace with the shrinkage in demand and by this means have managed to avoid any further fall in prices. The steel makers have not got quite as far as that. They have been fighting each other for so long in the market for certain classes of steel that they are more or less "down and out," so much so that in some quarters there appears to be a desire to get the makers together to try to repair the damage that has been done. To-day the trade in small steel bars, soft steel billets and thin sheets is not worth having at the prices which have been established by this senseless competition. It is no use saying that there was no alternative in face of continental competition. It has to be recognised at the outset that it is hopeless to try to meet the foreign prices. Nevertheless there is still a considerable share of the home trade for which British steel will be used, and it would have been far wiser to concentrate on this and at the same time, by intelligent co-operative effort, to maintain a reasonable level of price. But the selfishness of some makers—and the larger combines are the worst sinners in this respect—has prevented anything of that kind, and now business in the products we have just mentioned, soft billets at about £5 per ton, small bars at £6 15s. to £6 10s., and sheets at £8, can only be done at a loss. The steel makers are placing themselves in an unsatisfactory light to the outside world. They are weakening their already critical financial condition, they are arousing a feeling almost akin to contempt in the minds of their customers, and they are exposing to ridicule their scheme of control which they appear to exercise over the other portion of their production, i.e., plates and sections.

The Complaint of Inefficiency Unjustified

It is matters of this kind, and not the technical condition of the works, which give rise to the complaint of inefficiency which is levelled against the steel trade as a whole, and it is not without foundation. A typical instance is one to which reference has been made on previous occasions. The trade in round bars covers sizes from $\frac{1}{2}$ in. up to 9 in. diameter. Sizes 3 in. and over are controlled. Bars below 3 in. will be sold at £6 15s. by members of the steel associations, but for bars 3 in. to $5\frac{1}{2}$ in. these same makers must charge £9 7s. 6d. Above $5\frac{1}{2}$ in. there is a heavy extra list, which makes the price of 8 in. rounds £14 7s. 6d. Yet before the war the trade in these sizes was done with a maximum range in price of 20s. per ton between the $\frac{1}{2}$ in. and the 8 in. The steel makers now are trying to compete with the continental makers for the small sizes, yet at the same time they are driving the trade in the larger sizes out of the country. The bulk of the business

in large rounds over $5\frac{1}{2}$ in. has gradually passed to the Continent, where all sizes can be obtained with practically no extra charge; and as the basis price is considerably lower than the official home price in this country, a large share of the business in the smaller sizes has gone too. That could have been avoided to some extent by a judicious revision of the extra list, as originally the finish of the foreign large bars was not equal to the British, but since then the makers have had time and inducement to remedy the defect.

A similar position obtains in plates and sheets. The associated makers must charge not less than £9 12s. 6d. for sheets $\frac{3}{8}$ in. thick, yet they are selling $\frac{1}{4}$ in. sheets at £8. Tank or ship plates $\frac{3}{8}$ in. thick must be sold at not less than £8 17s. 6d., but boiler plates, which, as everyone knows, are more costly to produce, are offered at prices ranging from £8 15s. to £8 2s. 6d., and the crowning folly is the acceptance by one maker of a large order for boiler plates (which are to be made into boilers to be sent to Russia) at a price below £7 per ton! Is it any wonder that the cry goes up for the removal of the control of the industry from the hands of those who are responsible for such anomalies?

Controlled Material at Uncontrolled Prices

Another form of competition has now established itself, which is making the official control of the prices of plates and sections look stupid. If a consumer has a large order to place he knows quite well that he need not pay the official price for the steel. He can make a coupled bargain, by which he obtains some uncontrolled material at a special price as an inducement to place the order for the controlled material. True, that practice is contrary to the association regulations, but it is actually being carried out. But that is not the worst. Again, owing to the lack of understanding amongst the steel makers, merchants are allowed to sell the controlled products at whatever price they like, with the result that certain large merchant firms, who have an extensive connection in foreign steel, are offering to supply plates and sections from the British works at prices 1s. 6d. to 5s. per ton below the makers' own prices. They do this because they can recoup themselves out of the profits on the foreign steel and because it enables them to preserve their valuable trade connections with consumers in this country, who would otherwise do their business direct with the steel works.

Many definite instances of this could be given, the steel maker receiving the full price from the merchant, who gives the consumer a lower price. At one time much resentment was felt amongst consumers that they could not get lower prices direct from the works, but that has passed, and now they merely take the advantage that is offered to them. This means that the distribution of the orders is left to the merchant. The pig iron makers saw the danger of this and, in the Midlands, the merchant is not allowed to sell below the official price, but gets a standard commission. It would be a simple matter for the steel makers to make a similar provision; in fact, the North East Coast and the Scotch areas are already protected against underselling by merchants, but the Midland area is left at their mercy, no doubt because it suits some makers, who are getting the orders from the merchants. All these things call for a widening of the scope of the associations and a revision of their regulations, which in many respects are out of date.

Upward Movement to Prosperity in Sight

One cannot close this article without a reference to the political situation, which is bound to affect the iron and steel trade. A prompt and statesmanlike settlement of the financial crisis may lead to the restoration of a feeling of confidence in industry which will be the beginning of the upward movement to prosperity. The possibility of the adoption of protection has already aroused a more optimistic tone and the opinion is growing that the turn of the tide has come. We must hope that this proves true, and that the dismal experience of the past years will give place to a steady improvement in our industry, so that employers and workers alike will see a better return for their labours.

Some Inventions of the Month

By Our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature published weekly in THE CHEMICAL AGE.

Acid Inhibitors

MERCAPTANS of the type R-SH (in which R is a hydrocarbon radicle or anthraquinonyl, either of which may be substituted by one or more alkyl, alkoxy, or amino groups, but not otherwise) are used as acid inhibitors in metal-pickling baths and in the removal of rust without injury to a metal surface, especially in the removal of rust from the interior of piping. Specified mercaptans are thiophenol, thio-2-naphthol, benzyl mercaptan, 2-amino-5-ethoxythiophenol and 1-mercapto-2-aminoanthraquinone. They may be employed in conjunction with wetting or dispersing agents such as sulphite cellulose pitch, turkey red oil, Twitchell's reagent, aldehydenaphtholsulphonic acid condensation products, petroleum sludge acids, aldehydes, carbohydrates, etc. See Specification 346,992 (Imperial Chemical Industries, Ltd., London), dated April 15, 1930, a communication from E. I. du Pont de Nemours and Co., of Wilmington, Delaware, U.S.A.

Alloys

A HARD and highly-tenacious alloy described in Specification 348,641, dated February 10, 1930, by W. Müller and Olga, Prinzessin zur Lippe, of Berlin, consists of at least 55 per cent. of tungsten and/or molybdenum, preferably up to about 2 per cent. of carbon, and 26—40 per cent. of one or more additional metals of which at least 20 parts must be chromium if the tungsten-molybdenum content does not exceed 70 per cent. Other specified additional metals are iron, manganese, cobalt and nickel. Up to 3 per cent. of a hardening agent such as boron, silicon, or aluminium may be added. The alloys are prepared by heating the elementary constituents together so that they are completely molten for a short time.

CONTAINERS, pipes, boiler parts, and similar articles which are to be subjected to high temperatures and pressures, are made of steel alloys containing no aluminium, copper, or nickel, less than 0.2 per cent. of carbon, up to 0.8 per cent. of molybdenum and one or more of the elements chromium, tungsten, vanadium, silicon, and titanium. They are subjected to a heat-treatment of quenching and annealing. See Specification 348,668, International Convention date, December 24, 1928, by F. Krupp Akt.-Ges., of Essen, Germany.

STAINLESS iron-chromium alloys described in Specification 349,009, dated January 11, 1930, by F. Atkinson, of Totley Rise, Sheffield, and T. Hagon, of Beauchief, Sheffield, contain 14 per cent. or more of chromium, 0.08—0.25 per cent. of carbon, not more than 4 per cent. of nickel, a relatively small amount of copper (not substantially above 2 per cent.), and the remainder iron with the usual small proportions of silicon, phosphorus, sulphur, and manganese.

ALUMINIUM alloys, of which the hardness, elastic limit, impact resistance and machining properties improve on ageing, are obtained by starting with an alloy containing 1.25 per cent. of copper and approximately 0.1 per cent. of nickel, and when this is ready for casting, adding 0.2—5 per cent. of an alloy of magnesium with a heavy metal such as copper, nickel, zinc or tin. The finished alloy preferably contains 0.1—0.25 per cent. of magnesium. Manganese and up to 6 per cent. of iron, chromium, cobalt, tungsten, tin, vanadium, cadmium, silver, or titanium may also be present. Working during ageing does not impair the improvement in properties. The alloys are specially applicable for "shrinking on." See Specification 349,463, International Convention date, February 27, 1929, by Akt.-Ges. der Eisen-und-Stahlwerke vorm. G. Fischer, of Schaffhausen, Switzerland, and Messingwerk Schwarzwald Akt.-Ges., of Villingen, Germany.

Coating Metals

IN the production of carbonized metals, such as nickel or nickel alloy wire for use in electrodes of electron discharge devices, the surfaces of the metals are first oxidised and the oxidised surfaces are then exposed at elevated temperatures, e.g., at 800—1000° C., to an atmosphere of a hydrocarbon gas, such as acetylene containing a vaporised liquid hydrocarbon, e.g., petroleum, ether, naphtha, or kerosene. The carbonised metal is finally subjected to a degasification *in vacuo*. See Specification 347,267, International Convention date, January 25, 1929, by Westinghouse Lamp Co., Bloomfield, New Jersey, U.S.A., Assignees of C. V. Iredell, of East Orange, New Jersey, U.S.A.

Extracting Iron

To obtain spongy iron by the action of a reducing gas on the ores, a mixture of finely-divided ore and a binding agent is fed into a rotary drum to agglomerate it into balls, the latter are hardened by passage through a device heated only sufficiently to harden the binding agent without fusing the ore, the hardened balls are screened, and balls of equal size are fed in layers into the reduction furnace. Specified binding agents are aqueous solutions of magnesium chloride, of potassium silicate, and of sodium silicate. See Specification 348,033, International Convention date, February 8, 1929, by F. Krupp Akt.-Ges. Friedrich-Alfred-Hütte, of Rheinhäusen, Germany.

Extracting Tin

IN the treatment of materials containing tin in the form of oxide with solid, liquid, or gaseous reducing agents, e.g., in a rotary furnace, the temperature is maintained during the reduction below the melting point of the charge and is subsequently raised sufficiently to produce a sintered or pasty slag which envelops the reduced tin. An addition to the charge of acid or basic materials or of coke or coal is made before, during, or after the reduction in order to adjust its composition to yield the required slag. The tin is recovered from the slag by known methods. See Specification 349,313, dated May 29, 1930, by Berzelius Metallhütten Ges. of Duisburg, M. G. Freise, of Angermund, near Düsseldorf, and H. Maschmeyer, of Bad Ems, Germany.

Heat Treatment of Metals

A FUSED salt bath suitable for use in hardening high-speed tool steels at 1700—2400° F. (927—1316° C.) comprises a metal (e.g., steel or chrome iron) container in which the salt is heated by a low voltage current passed between one or more centrally immersed electrodes (e.g., of steel or chrome iron) and the container wall. The initial fusion of the salt is effected by means of an arc or by ignition thereon of a compound of the "Thermit" (Registered Trade Mark) type. The articles under treatment may be removed when no longer magnetically attracted by the electrode. See Specification 347,238, International Convention date, December 14, 1928, by A. E. Bellis, of Branford, Connecticut, U.S.A.

Treating Copper Ores

ORES, concentrates, etc., containing copper in the form of sulphate, are heated with a carbonaceous material and a segregation reagent such as a halogen or halogen compound. The copper is thus segregated from the ore particles while remaining admixed with the charge, and may be recovered by froth flotation. Ores containing copper-sulphide with or without oxide or sulphate are subjected to a preliminary roasting to convert the sulphide into sulphate. See Specification 348,024, dated January 21, 1930, by Minerals Separation, Ltd., of London, and T. J. Taplin, of Kew, Surrey.

Operating Blast Furnaces

THE charge for a blast furnace is made up with a flux such as limestone or dolomite which has been roasted without loss of the size and strength of the particles or lumps beyond the minimum required to support the load in the furnace. Preferably the roasting kiln is close to the furnace in order to avoid the injurious effect of the atmosphere as the flux hot from the kiln is charged in a partly burnt condition to the furnace. To ensure a uniform content of base such as lime in the charge irrespective of the degree of burning of the flux, the latter is measured by volume for making up the charge. See Specification 349,015, International Convention date, February 11, 1929, by E. Baumgartner, of Kladno, Czechoslovakia.

Refining Metals

IN a refining process described in Specification 349,446, International Convention date, February 20, 1929, by G. H. Clamer, of Atlantic City, New Jersey, U.S.A., a charge of the metal is poured into a furnace or ladle disposed within an induction coil supplied with current at commercial frequency and the ampere-turns of the coil are so adjusted that while the heat supplied to the bath is merely sufficient to maintain the temperature of the charge, the stirring effect causes slag and oxidation products to accumulate at the sides of the bath for ready removal at intervals. A jet of air or gas may be directed on to the crown formed centrally of the bath. The slag layer may be heated by arcs receiving current from a polyphase system. The axis of the coil is preferably adjustable for regulating the stirring effect.

Monthly Metallurgical Section

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NOTICE.—Communications relating to editorial matter for our *Monthly Metallurgical Section* should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154, Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

Desulphurisation of Cast Iron by Soda Ash **Effects of Sulphur as Impurity in Cast Iron**

This article outlines the advantages to be gained by the use of soda ash in the desulphurisation of cast iron. It is based on investigations carried out by the metallurgical research staff of Imperial Chemical Industries, Ltd., and was recently the subject of several brochures.

THE control of the sulphur content of cast iron is probably the most important problem encountered in modern foundry practice. The harmful effect of sulphur as an impurity in the metal is now generally recognised and there is an increasing tendency to lay down stringent specifications which demand a low sulphur content. Even when the amount of sulphur contained in the pig iron is low, serious difficulty is experienced in satisfying the requirements of modern specifications by reason of the increased sulphur content of the finished castings which results from the use of scrap metal and the introduction of sulphur from the coke in the cupola. In order to ensure that the sulphur content will not exceed the low figure demanded, it is necessary to pay careful attention to the quality of the coke used and to limit the proportion of scrap which is remelted in the production of the metal.

The problem is of special importance when the metal is required for conversion into steel by the Tropenas converter because a 20 per cent. concentration occurs during the conversion and the sulphur content is increased in the same proportion. In order that the amount of sulphur in the finished steel castings shall not exceed 0.06 per cent., which may be regarded as a desirable maximum figure, it is necessary to use pig iron of a very low sulphur content, high grade metallurgical coke and only very small proportions of scrap metal. The restrictions which are thus imposed on the foundry in regard to the choice of raw materials must necessarily increase to a considerable degree the cost of manufacture of the final product. Thus it is well-known that cokes of low sulphur content are obtainable only from coal which occurs in certain areas, and foundries situated in other districts must therefore be placed at a serious disadvantage if the demands made upon them are such that high grade cokes must be used in order to maintain the quality required in their finished castings.

It is evident, therefore, that the difficulties indicated could be to a great extent overcome by the introduction of a cheap, reliable and efficient method of desulphurisation of the metal during the process of manufacture. A method which satisfies these requirements and is based on the use of soda ash as a desulphurising agent is now available, and it has been conclusively established that its adoption results in a marked reduction in the sulphur content of the metal. It is now possible by the use of a relatively small amount of a cheap industrial chemical not only to reduce the sulphur content of castings produced from the raw materials normally used, but also to cheapen the cost of production of castings of high quality by enabling the manufacturer to use pig iron and coke of greater sulphur content and to mix a larger proportion of scrap metal in his melt.

Soda Ash as a Desulphurising Agent

As early as 1867 Horsley, of Derby, called attention to the effect of the addition of alkalis in removing sulphur from molten iron and since that time numerous proprietary products have been marketed as desulphurising agents. These products, which are usually sold in the form of blocks or bricks, always contain considerable proportions of alkaline ingredients, and it is reasonable to assume that their action on the sulphur present in the metal is associated with their alkaline properties. As no systematic study of the action of alkalis on molten iron appeared to have been made it was decided by Imperial Chemical Industries that the question

should be the subject of an exhaustive investigation by their metallurgical research staff. The results obtained in this investigation proved to be of the greatest interest, and it has been conclusively demonstrated that a 50-60 per cent. reduction in the sulphur content of the metal can be effected by the use of relatively small proportions of soda ash.

In ordinary foundry practice the desulphurisation is effected by placing the required amount of dry soda on the bottom of the ladle and then tapping out the metal from the cupola at as high a temperature as possible and preferably at 1,400-1,450° C. A vigorous but not violent interaction between the metal and the alkali takes place with rapid evolution of carbon dioxide and formation of sodium sulphide and other sulphur compounds. The vigorous evolution of carbon dioxide maintains the molten mass in a state of agitation and ensures intimate contact between the metal and the alkali reagent. When the molten metal becomes quiescent a thin soda slag, which contains sodium sulphide and other sulphur compounds, collects on the surface and in cases where the cupola is tapped intermittently the subsequent batches of metal pass through the slag and thus cause further agitation, which increases the efficiency of the removal of the sulphur. When the ladle is full a quantity of ground limestone is thrown on to the slag and rabbled into it. The limestone absorbs the thin alkaline layer and causes the formation of a thick slag which can be raked off without difficulty. The thickened slag should be removed as completely as possible and the desulphurised iron may then be cast in the usual way or run into the converter if it is required for the making of steel.

Quantity of Soda Ash Required

It has been shown that an amount of soda ash equal to about 1 per cent. of the weight of the metal or about 20 lb. per ton is sufficient in ordinary cases and that the amount of ground limestone required for the thickening of the slag is about one-half the weight of soda ash used. The total cost of the materials used is approximately 1s. 6d. per ton of metal and the process of desulphurisation necessitates no important variation in the normal foundry routine.

The effect of the use of soda ash on the refractory linings of the ladles has been considered in the course of the investigation of the desulphurising process. It is known that siliceous refractories are attacked to an appreciable extent on prolonged exposure to slags containing sodium compounds. No difficulty from this cause will arise in foundries where ladles of capacity not more than two tons are used, because it is customary in such cases to line the ladle with a ganister daub which is knocked out after the day's run. When large brick-lined ladles are employed the bricks used should be made from Scotch fireclay and they should have a close even texture and show good resistance to spalling. The joints should be as thin as possible and a protective graphite wash may be applied to the surface of the brick lining as an additional precaution against the slight corrosive action of the slag.

Effect on Quality of the Metal

This process of desulphurisation was originally based on a small-scale laboratory investigation in which 14 lb. melts were made in a high-frequency induction furnace, and the effect on the efficiency of the process of various factors such as the original sulphur content of the metal, the proportion of soda ash used, the time of contact of the metal with the desulphurising agent, etc., were studied.

The conclusions reached were that (1) when the amount of sulphur in the original charge lies between 0.13 per cent. and 0.60 per cent., and when a constant amount of soda ash is used, the reduction in sulphur content is greater with the higher percentage of sulphur present before desulphurisation; (2) between the limits of 0.225 per cent. and 1.5 per cent. of soda ash in the weight of metal taken, the reduction in sulphur content is approximately proportional to the amount of soda ash used; (3) the extent to which sulphur is removed can be considerably increased by repeating the soda ash treatment after the first slag has been thickened and separated in the usual manner (this procedure was shown to result in a reduction of the sulphur content by nearly 80 per cent., and its adoption in practice may be justified when the castings must conform to the requirements of a very rigid specification); (4) the manganese content is not appreciably affected by the desulphurisation process; and (5) the process improves the physical properties of the metal, in that the alkali reagent has a cleansing and degasifying action, and greatly reduces the number of non-metallic inclusions in the finished castings.

Use in Making Steel Castings by Tropenas Process

The steel scrap and high silicon pig iron were melted in a cupola and the molten iron was desulphurised by means of soda ash in receiving ladles of 30 cwt. capacity. The desulphurised metal, after the removal of the slag, was transferred to the converter and blown into steel by the usual procedure. Samples of the metal leaving the cupola, of the desulphurised iron and of the finished steel were taken and their sulphur content was determined by analysis, the following results being recorded:—

Sample No.	Description of Sample.	Reduction in	
		Sulphur Content.	Sulphur Content.
		%	%
1	Cast iron. First heat, before desulphurising.....	0.079	
2	Cast iron. First heat after desulphurising.....	0.033	57
3	Finished steel. First heat.....	0.036	
4	Cast iron. Second heat before desulphurising.....	0.087	
5	Cast iron. Second heat after desulphurising.....	0.035	60
6	Finished steel. Second heat.....	0.039	

These results show that a reduction of 57 and 60 per cent. in the sulphur content of the iron was effected by the use of soda ash. The slightly increased amount of sulphur contained in the finished steel compared with that present in the desulphurised iron is accounted for by concentration in the converter. At the time when the trial was carried out the sulphur content of the finished metal had increased to the high figure of 0.09 per cent. and difficulties had arisen as a result of the cracking of the steel castings. By the adoption of the soda ash desulphurisation process the amount of sulphur present in the finished castings was reduced to the low figure of 0.04 per cent., which satisfies the most stringent requirements. The process is now in regular use at the foundry where these trial experiments were made.

Desulphurisation in the Iron Foundry

In a series of trials carried out in an iron foundry the charge to the cupola consisted of one part of pig iron and two parts of best machinery scrap. The metal was tapped into a 30 cwt. ladle, about 10 cwt. at a time. Desulphurisation was effected in the ladle by the procedure previously described, special test bars and ordinary castings being made for examination. The results of the analyses of samples of the metal are given below:—

Sample No.	Composition of Metal.			Soda Ash used.
	Sulphur.	Silicon.	Manganese.	
	%	%	%	%
1	0.14	2.35	0.54	Nil.
2	0.13	2.18	0.54	Nil.
3	0.062	1.93	0.53	About 1
4	0.054	2.00	0.54	About 1
5	0.058	1.98	0.52	About 1

These results show a reduction of 50 to 60 per cent. in the sulphur content. The silicon content is slightly reduced and the manganese content is not appreciably affected. The physical properties of the metal were investigated, and it was shown that desulphurisation had resulted in a marked improvement in the compression, transverse and tensile

strengths. Microscopic examination of the desulphurised metal showed that it was nearly entirely free from non-metallic inclusions, and that the treatment with soda ash had resulted in a very marked improvement in this respect.

Beneficial Effects of the Process

One of the advantages to the manufacturers of iron and steel products which result from the simple and inexpensive process of desulphurisation by soda ash is that the process is simple in operation and involves no important changes in the normal foundry routine. It can be applied at various stages in the production of iron and steel products. The sulphur content of the metal can be reduced by about 50 to 60 per cent. by the use of soda ash in the proportion of about 20 lb. per ton of metal, and the cracking of steel castings which frequently results from the presence of a high proportion of sulphur in the metal can be prevented.

The reduction in sulphur content, moreover, is accompanied by a definite improvement in the physical properties of the metal, and by the almost complete elimination of non-metallic inclusions of sulphides, oxides, etc. This effect is of special importance in foundries producing castings which are to be submitted to a hydraulic test or which are exposed in use to the action of corrosive liquors. The desulphurisation process also enables the manufacturer to obtain products of improved quality from the raw materials in normal use or to maintain the standard quality of his products, while at the same time using raw materials of higher sulphur content. Cost of manufacture is thus reduced because products which would normally demand the use of high quality raw materials can be made by the use of pig iron and coke of higher sulphur content, and a greater proportion of metal scrap.

Calcium Carbide in Iron Smelting

Successful Use in Preparation of Perlit

CALCIUM carbide enters into a large number of flux compositions which, however, seldom act so effectively as when the constituents themselves are added in accordance with the approved molecular formula. Used alone, however, it has been successfully employed in the preparation of "Perlit," which is an iron of high combined carbon content. Various methods have been employed to make Perlit iron, and the carbide is only used when difficulty is experienced in raising the percentage of combined carbon. There are several reasons for this, but the main consideration is the cost of raw materials. Pig irons rich in combined carbon are rarer and dearer than those of low carbon content. Scrap iron is invariably diluted, and in general may be regarded as a low combined carbon alloy. It is of course the cheapest form of iron to be had which allows of a margin for other more expensive constituents to be added.

Method of Adding the Carbide

In working the process the bogie into which the iron is to be tapped is previously prepared. Calcium carbide if placed in the hot bottom of the receptacle would only lose gas to no purpose, whilst on the other hand cold material of any description would risk the possibility of explosion. To circumvent those difficulties the weighed proportion of carbide is placed on a thin bevelled sheet of iron a few moments prior to tapping. This allows it to be moderately heated up by radiation from the walls and bottom of the bogie. The tap hole of the cupola is then opened and the molten iron run into the bogie. The metal does not land on the surface of the carbide in the first instance, if properly tapped, but swirls underneath the bevelled sheet and rises over the carbide. Despite these precautions, however, quite a proportion of the salt scatters to the accompaniment of a crackling sound and flashes of light from the ignited acetylene. The loss of carbide sustained is very obviously a high one, but what acetylene reacts with the iron directly adds the required carbon. The average loss from several sets of bogies is not, however, so widely divergent, and by a system of mixing the ultimate carbon figure is brought close to specification.

Manganese has also to be added together with the carbide since scrap is usually low in this element and would not assist in the formation of manganese carbide. The iron has to be tapped at a slightly higher temperature than for ordinary castings as the ferro-manganese has to be dissolved. This means that greater heat than usual is applied to the carbide and the loss sustained is accordingly higher.

Metallurgical Topics: Monthly Notes and Comments

From Our Own Correspondents

Conditions in Iron and Steel Industry

THE changed conditions in international finance have transformed the outlook in the iron and steel industry. The most significant factor is the sudden spurt of inquiry from abroad, where consumers of pig iron are exploring the advantages of securing British material, now that in terms of foreign currencies it is nearly 20 per cent. cheaper. The price of East Coast hematite has risen by 3s. 6d. a ton. Business has been done at the new quotation of 63s., and it is freely suggested that the price will advance to 65s. in the near future. Quotations of Cleveland iron have not been advanced, but they are much firmer, and lately fixed prices have not been at all closely observed. In the steel trade prices remain unchanged, but there has been a welcome improvement in inquiry, principally from overseas. Towards the close of last week imported semi-finished steel from the Continent was marked up £1 per ton, equal to a rise of about 25 per cent. At the moment there is no prospect of more plant being put into commission, for existing production has been considerably in excess of current requirements, and has caused a large accumulation of pig iron. So far the depreciation of sterling has not had any effect upon imported ores, for the reason that no business is being done. All the ironmasters of the district have large stocks of ore.

The Metallurgy of Tinplate

THE autumn meeting of the Iron and Steel Institute began on Tuesday, at Swansea, the seat of the tinplate industry, one of the branches of the iron and steel trades which, like certain sections of the light castings industry, has suffered less during the present depression, than the heavier branches. It was appropriate therefore that some of the papers down for reading should relate to the industry, or, at least, to some of the scientific aspects of the manufacture of tinplate. Indeed, out of the only four really practical papers in a programme of thirteen altogether, three were devoted to this subject. They were on "Mottled Tinplates," by Mr. J. C. Jones, M.Sc.; one by Professor Bannister and Mr. R. D. Jones, on "The Diffusion of Tin into Iron, with Special Reference to the Formation of Columnar Crystals," and one by Professor C. A. Edwards, D.Sc., F.R.S., and Mr. A. Preece, M.Sc., on "A Study of the Constitution of the Iron-Tin Alloys." The latter establishes the existence of three iron-tin compounds: Fe_2Sn , which is stable between 760°C . and 900°C ., but can react with tin at 800°C ., with the production of FeSn ; FeSn itself, stable at all temperatures below 800°C ., but reacting with tin below 496°C . to produce FeSn_2 ; and lastly, FeSn_2 , which exists below 496°C ., and when heated to above that temperature breaks down into FeSn , plus a tin-rich liquid. No evidence was found of the existence of Fe_3Sn , and the authors regard the solubility of iron in solid tin as extremely low, while that of tin in solid iron increases with the temperature up to 760°C ., becoming less as the melting point of iron is approached.

"Mottled" Tinplates

MOTTLED tinplates are, according to Mr. J. C. Jones, a comparatively recently encountered defect, and one the occurrence of which he attributes to (1) the steel base, and (2) the tinning-pot conditions. Electrolytic iron, tinned under precisely similar conditions as the mild steels ordinarily used, showed hardly any mottling, whereas the steel sheets did. Tin-bar steel imported from the continent enjoys an enviable reputation, says the author, in respect of being free from this defect. This would seem to lend additional support to the belief held by a good many tinplaters, that basic Bessemer steel is better, as a basis, than openhearth steel, a belief mentioned in the discussion in May last, before the Iron and Steel Institute, of the paper by Mr. Vernon Harbord on the possibility of reviving the Bessemer steel industry in Great Britain. Mr. Jones also found that if the zinc flux used in tinning becomes stale the defect occurs, while adding new flux removes it. Aluminium chloride, substituted for zinc chloride, gave excellent results, and bright, unmottled plates resulted from its use. The experiments described in the paper by Professor Bannister and Mr. R. D. Jones were carried out on Armco iron, and the rationale of the diffusion of tin into iron studied.

The mode by which, and the depth to which, the tin diffused, were both investigated; the latter is very small, but on heating tin and iron together it was found that columnar crystals of ferrite are formed at the surface, whereas if the iron is heated alone this does not happen. The degree of diffusion and the columnar formation seem to be related.

Light Alloys in Heavy Engineering

THE light alloys, such as duralumin and alpac, have already been extensively employed for transport vehicles, and have redeemed their initial cost by the decrease in power consumption required to propel them. A more novel application of light metals is, however, in heavy engineering, and, in particular, in the construction of overhead cranes. A recent issue of the *Revue de l'Aluminium* gives a description of a ten-ton overhead crane built up of light metal sections and castings, recently installed at the Alcoa Engineering Works at Massena. Other examples are afforded by the use of aluminium for the construction of mine and colliery cages, and at Haussham, near Munich, where such cages have recently been introduced, the general saving in time and power has had the result of increasing by no less than 50 per cent. the output of the mines. The most novel application of all is, however, the substitution of light alloys for the heavy iron castings and framework of electric lifting magnets. These are now in use at a steel works in the St. Etienne district, where they are employed not only for lifting ingot moulds, but in the scrap-yard, for the handling of iron and steel scraps.

Metallurgical Research at N.P.L.

THE research programme of the Metallurgical Department of the National Physical Laboratory at the present time comprises work on alloys of iron for the Alloys of Iron Research Committee, which includes representatives of the great technical institutions, and on aluminium and other light alloys for the Metallurgy Research Board, the latter being of special importance to the Air Ministry and the Aeronautical Research Committee; fundamental research into the physical constitution, structure and properties of metals and alloys; investigation of some fundamental problems connected with the presence of gases in steel; the study of refractory materials capable of withstanding high temperatures; research on dental alloys and amalgams; investigation of the causes of cracking of boiler plates, and much other work. The Department is also co-operating in the researches on fatigue phenomena, and in the investigation of alloys at high temperatures, spring steels and wrought iron chains and other lifting tackle to which reference has already been made in the section dealing with the Engineering Department. In addition investigations are in progress for the British Non-Ferrous Metals Research Association. Special investigations are being undertaken by the Department for firms and other bodies to elucidate, and, if possible, find remedies for, difficulties occurring in engineering and manufacturing practice. Under an arrangement with the Iron and Steel Institute, standard steel samples of carefully determined composition have also been prepared and can be purchased, for the control of chemical analyses, where required.

Copper Production in Canada

THE rapid expansion in the production of copper in Canada in recent years is again exemplified by the Final Report of the Dominion Bureau of Statistics at Ottawa on output in 1930. The production of copper last year in Canada is shown to have reached the record total of 303,478,356 lbs., valued at \$37,948,359, as compared with 248,120,760 lbs., valued at \$43,415,251 in 1929. This was an increase of 22 per cent. in quantity, but a decrease of 12.5 per cent. in value owing to the extremely low price of copper ruling in 1930. Increased productions realised in Ontario and Quebec reflected the expansion in the mining and metallurgical operations of the International Nickel and Noranda Copper producing companies. A production of 93,318,885 pounds of copper in British Columbia was considerably less than that of the previous year. Commencement of production of electrolytic copper in Eastern Canada by the Ontario Refining Co., Ltd., at their new plant at Copper Cliff, Ontario, was an outstanding event in the Canadian copper industry during 1930.

Some Inventions of the Month

By Our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature published weekly in THE CHEMICAL AGE.

Alloys

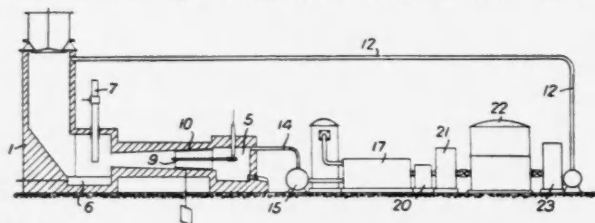
AN alloy possessing valuable antifrictional qualities, and having a structure of solid solution and eutectoid, consists of 62 per cent. of copper, 30 per cent. of zinc, 4 per cent. of tin, 2.5 per cent. of lead, and 1.5 per cent. of nickel, according to Specification 349,484, International Convention date, February 1, 1929, by H. Kanz, of Zurich, Switzerland.

Aluminium and its Alloys

THE electrothermic reduction of alumina-containing materials is effected in the presence of iron, silicon, or other substance capable of reducing the vapour pressure of the aluminium, and the resulting crude alloy is thereafter separated into an alloy rich in aluminium and another alloy poor in aluminium by known processes such as liquation, centrifugation and cooling. When iron is used the crude alloy must contain at least 70 per cent. of aluminium. Aluminium may be obtained from the rich alloy by distillation *in vacuo*. Methods for the treatment of clay, kaolin, white bauxite, and red bauxite are described in detail. The process is suitable for the production of aluminium-silicon alloys by reducing materials poor in iron oxide, *e.g.*, kaolin. See Specification 351,524, March 24, 1930, by W. Neumann, of Lausitz, Germany.

Magnesium

THE production of magnesium by reduction of its compounds with charcoal in an electric arc, in a current of hydrogen or other inert gas, is described in Specification 351,295, International Convention date, August 3, 1929, by Oesterreichisch Amerikanische Magnesit Akt.-Ges., of Radenthein, Carinthia,



351,295

Austria. A mixture of magnesite and charcoal is fed to the arc 6, 7, and the resulting mist containing metallic magnesium passes at 650–1,200° C. between a rod cathode 9 and an annular anode 10 of an electrostatic separator. Liquid magnesium collects in the receiver 5, and the gases pass through a washer 15 or through a second electrostatic separator to a regenerator 17 in which carbon dioxide and hydrogen are formed by reaction of carbon monoxide with steam. After compression at 20, the carbon dioxide is removed by a washer 21 and the hydrogen is recirculated after passing through a drier 23.

Nickel

A BLAST of superheated steam with or without admixture of a small proportion of air is employed for Bessemerising nickel-containing matter according to Specification 349,700 (Wade), dated March 28, 1930, a communication from International Nickel Co., Inc., of New York. The temperature of the bath may subsequently be raised and the material be blown with superheated steam alone in order to eliminate substantially all the sulphur. In an example the sulphur content is reduced from 18 per cent. to 2–4 per cent. in the first stage at 2,450–2,650° F. and further reduced to 0.005–0.02 per cent. in the second stage at 2,750–2,850° F.

Reducing Ores

IN a process for recovering sulphur, copper and arsenic from pyritic ores the ore is smelted in a blast furnace with fluxes and with solid reducing agents in such proportion that in the lower part of the furnace a pyritic or a partial pyritic smelting occurs, while in the higher part the reducing agent is substantially consumed by the sulphur dioxide with or without arsenious oxide and oxygen with formation of elementary sulphur with or without arsenic. The sulphur and arsenic so produced are withdrawn and condensed. The greater part of the iron is oxidised in the lower part of the furnace as well as the sulphur and forms a slag with the fluxes,

while the sulphur not burnt forms a matte with the unoxidised iron and any copper present. The air supply at the tuyères may be mixed with the sulphur dioxide produced by roasting the matte from the furnace or by roasting any ore too fine for direct smelting. See Specification 350,625 (Mellersh-Jackson), dated March 31, 1930, a communication from Patentaktiebolaget Grondal-Ramen, of Stockholm, Sweden.

Refining Lead and Lead Alloys

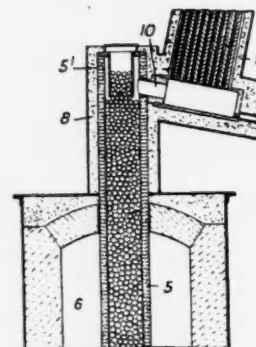
LEAD containing one or more of the oxides of lead, arsenic, tin and antimony or one or more of the elements arsenic, tin and antimony, with or without sulphur or copper, is refined by heating above the melting point in presence of a reducing agent such as bituminous coal and a flux comprising stable oxygen-containing salts of the alkali metals and a fluoride, *e.g.*, a mixture of sodium carbonate, borax, and fluorspar. In addition, the molten material may be blown with a gas containing oxygen. The product may be a lead-antimony-tin alloy or the tin and antimony may be removed successively, leaving the lead substantially pure. The process is especially applicable to the treatment of lead battery plates. See Specification 349,607, dated February 28, 1930, by C. R. Hayward, of Quincy, Massachusetts, U.S.A.

Treating Nickel-Copper Mattes, etc.

A METHOD for recovering nickel and copper with or without other metal values from sulphide materials, especially mattes, containing them, is described in Specification 351,150, International Convention date, August 17, 1929, by International Nickel Co., Inc., of New York, assignees of R. C. Stanley, of New York. The material is heated in presence of a stratifying substance to melt it and to resolve the mass into copper-bearing "tops" and nickel-bearing "bottoms," whereupon the "tops" and "bottoms" are separated, either in the molten state or after solidification, and are treated by dry methods for recovery of the copper and nickel. The matte may be smelted with sodium sulphide or a mixture of salt-cake and coke or calcium sulphide and the smelting may be effected in two or more steps. The "tops" may be blown to blister copper and the crushed "bottoms" may be roasted to obtain an impure nickel oxide, from which is obtained by reduction nickel containing small amounts of iron, copper, cobalt, silver, gold and platinum metals.

Zinc

THE gaseous products from a zinc smelting plant are passed through a suitable medium maintained at 800–900° C. to effect the removal of lead and other undesired metals such as antimony, tin, bismuth, germanium, thallium, aluminium and magnesium from the zinc vapours. The medium may be a residue or refractory contained in a chamber separate from



352,005

the smelting chamber, but it is preferred to use one or all of the ingredients of the charge in crushed or briquetted form, or aggregated by sintering. In the preferred arrangement shown the charge is introduced at the top of a retort 5 and worked off charge is removed at the bottom, the working being continuous. The smelting is effected in the lower part within the furnace 6 and the gaseous products pass through the cooler portion of the charge in the upper part 5, outside the furnace. The zinc vapour passes through an offtake 10 to a condenser 11. See Specification 352,005 (Mellersh-Jackson), dated February 3, 1930, a communication from New Jersey Zinc Co., of New York.

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NOTICE.—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, Bouverie House, 154 Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

Beryllium as a New Industrial Metal

By J. Becker

The following article, reprinted from the October issue of "Engineering Progress," outlines the present industrial use of commercial beryllium, which is a metal possessing new and very distinctive properties anticipating a demand for it when its production has been cheapened.

BERYLLIUM, which has been prepared commercially only within the last two years, is rapidly growing in industrial importance. It was first prepared in a pure state in 1828 by Wöhler, who was also the first to succeed in preparing pure aluminium in 1827. Its specific gravity is 1.84 (0.065 lb. per cu. in.). Apart from the alkali metals, it is therefore the lightest of all metals, and is one-third lighter than aluminium. Chemically it has an extraordinary affinity for oxygen, is harder than glass, and has its melting point at 1,280° C. These properties made it likely from the outset that beryllium would be of importance for industrial purposes, provided that it could be prepared in compact pieces.

Electrolytic Production

No process, whether of a chemical, thermal, or electrochemical nature, succeeded during the last century in producing pure beryllium otherwise than in the shape of flakes. It was not until 1921 that Stock and Goldschmidt recognised that in order to prepare beryllium in large pieces electrolytically the process must be carried out so that the temperature of the electrolyte lies above the melting point of the metal itself. By the use of suitable compounds of beryllium it was found possible to attain this temperature of the electrolyte by means of the electrolytic current and to maintain it. The process was taken over by the Siemens and Halske A.G., of Berlin, which further developed and perfected it for use on an industrial scale.

The electrolytical process of recovery demands a good deal of preliminary work in heating up the apparatus and the electrolyte, while the consumption of current is high and the crucibles are soon worn out at the high temperatures involved. These facts will serve to explain why the price for beryllium is at the present time still rather high, although it has dropped quite considerably since industrial production started. There is hardly any doubt that the fall in price will keep pace with the increase in the consumption of beryllium. The commercial product contains 98 per cent. beryllium and 1 per cent. iron, while the remainder is dross; it is possible, however, to produce beryllium in an almost pure state with a content of 99.5 per cent. beryllium.

Transparency to X-Rays

Even to-day there are numerous purposes to which beryllium is applied. The pure metal, it is true, is very brittle, so that it is difficult to work, but it can be pressed at red heat to thin plates of about 20 mm. in diameter and 1 to 2 mm. thick. These are used as windows in X-Ray tubes that are protected from radiation. Owing to its low atomic weight, beryllium allows X-Rays to pass through seventeen times better than aluminium, which was formerly used for these windows. If suitable processes should be found for working pure beryllium, there can be no doubt that its industrial importance will rise for purposes where an extreme saving of weight is desirable, but at present it is still comparatively slight. The commercial product is used almost exclusively for alloys.

Owing to its extraordinary affinity for oxygen, beryllium exerts a powerful reducing action and it is replacing phosphorus more and more for the reduction of cast copper. From a foundry point of view, cast copper reduced with beryllium has the same excellent properties as when reduced with phosphorus,

but it has a still higher density and especially a very much greater electrical conductivity. While it is hardly possible in the case of the phosphorus casting to exceed a conductivity of 44 (that of electrolytic copper being 60), a conductivity of as high as 56 can be attained with a copper casting that has been reduced with beryllium, a conductivity, that is to say, that corresponds to that of rolled electrolytic copper. Thus the size of cast parts of electrical plants can be reduced by 25 per cent., which means a great saving of material and a reduction in weight that is in many cases very much to be desired.

Heat Treatment of Beryllium Alloys

The most important beryllium alloys are those formed with copper, cobalt, and nickel. In all the beryllium alloys that are employed for industrial purposes, the beryllium content does not rise above 3 per cent., so that the high price of the metal does not stand in the way of their use. The strength of beryllium alloys can be improved by heat treatment and can be converted by a process of quenching and tempering from their original soft condition into a spring temper state, which is very valuable for technical purposes. This property of toughening under heat treatment is shown by beryllium bronzes with a beryllium content ranging between 1.3 per cent. and from 4 to 5 per cent.; it is most marked at a beryllium content of from 2.4 to 3.5 per cent. The alloy is heated for some time to about 800° C. and then quenched in water, whereby its soft condition is retained, so that it can be rolled, for example. If the alloy is afterwards tempered at temperatures of up to 400°, it hardens. The degree of hardness attainable depends upon the beryllium content of the alloy, the previous treatment, e.g., the extent to which it has been rolled, and the temperature and duration of the tempering process.

Beryllium bronze—as distinguished from beryllium-copper—differs greatly in its physical properties from other bronzes and is superior to them. A bronze containing 2.5 per cent. beryllium, without heat treatment, has a hardness after quenching of about 100 Brinell units, which can be raised to about 400 Brinell units by heat treatment. A toughened bronze containing 2.5 per cent. beryllium is far stronger than aluminium bronze double-rolled to spring hardness and approaches the strength of good spring steel. By varying the heat treatment, either an extraordinarily high strength with low elongation or a comparatively high strength with great elongation can be arrived at. Beryllium bronze has a high elasticity and shows extremely little sign of fatigue: in an endurance test, a helical spring of beryllium bronze withstood twenty-five million vibrations without losing any of its spring power, while springs made of phosphor bronze usually do not take more than a few hundred thousand vibrations. Beryllium bronzes resist corrosive action just as well as tin bronzes, but are very much more resistant to corrosion than steel. The electrical conductivity of 2.5 per cent. beryllium bronze is about 17 to 19, against 7 to 10 of phosphor bronze, so that structural parts of electrical equipment can be made about half the size when beryllium bronze is used. The thermal conductivity of beryllium bronze is 0.4, the figure for electrolytic copper being taken as 1. Beryllium bronze is thus an industrial metal which combines great strength with a comparatively high thermal conductivity.

An Alternative to Steel

The uses to which beryllium bronzes can be put industrially correspond to these properties. They can be used to advantage wherever the saving of material and in weight of current carrying parts justifies the higher price of beryllium bronze, or where their greater hardness and strength determine their employment in parts that are subjected to mechanical strain. It is advisable to use beryllium bronze especially for springs and small constructional parts the price of which is of no great moment in comparison with the price of the whole machine, but which are of particular importance for its reliable operation. In such cases, the higher initial cost is more than offset by the improvement in reliability of operation. Contact tins and brush-holder springs for electromotors, for instance, and highly-stressed springs in telephone and signalling installations, also moulds for casting certain kinds of ceramic material are nowadays often made of beryllium bronze.

Steel can profitably be replaced by beryllium bronze when there is danger of rusting or it is desired to avoid magnetising. The field of application in which beryllium bronzes can be used to advantage cannot be even approximately delineated at the present time, because we have here to do with a material the properties of which can be varied to an extraordinary extent according to the beryllium content of the alloy and the nature of its treatment. Recent investigations, for instance, have shown that beryllium bronze with a content of only from 0.9 to 1.5 per cent. beryllium is excellently adapted for use as a bearing metal. Bearing bushes of this material showed only about one-sixth of the wear of tin bronze in endurance tests, without the shaft being attacked.

Zirconium Alloys

Use in the Production of Steel Castings

INFORMATION concerning the use of zirconium in the production of alloys is contained in the United States Bureau of Mines, Information Circular No. 6455. Several hundred tons of zirconium ore are used annually in the production of zirconium alloys, which are produced in electrical furnaces and employed principally in the production of steel castings. When added to steel, zirconium is claimed to perform three important functions. By its powerful deoxidising action it vigorously reduces metallic oxides and scavenges non-metallic inclusions, but it does not tend to remain in the steel in the form of an oxide, as does aluminium. Secondly, zirconium combines with dissolved nitrogen, the greater part of the nitride being removed in the slag. It forms a malleable sulphide and diminishes hot-shortness of high-sulphur steel to a greater extent than manganese, although for economic reasons it cannot displace manganese entirely in the production of high-sulphur steels.

A zirconium treated carbon steel is therefore characterised by freedom from inclusions, which would act as nuclei for shock and fatigue failures; excellent hot working properties; and when suitably heat treated, tensile properties closely resembling those of special alloy steels, ductility being specially favoured by zirconium treatment. The alloy generally used is zirconium-ferrosilicon, which contains 13 to 14 per cent. of zirconium and 45 per cent. of silicon. Where a large excess of silicon would be undesirable, silicon-zirconium, containing 35 to 40 per cent. of zirconium, is used. Zirconium-nickel alloys have been patented for use in the manufacture of edge tools, especially machine tools for milling cutters and cast tools for lathes and planes.

Nickel Cast Iron in Motor Industry

AN interesting brochure on the use of nickel cast iron in the motor industry has been issued by the Bureau of Information on Nickel (Mond Nickel Co., Ltd.) It is stated that the addition of nickel to cast iron assists the designer by giving increased strength and hardness throughout castings of varying section, together with uniformity of structure and properties in all sections; by giving reduction of porosity troubles at difficult spots in casting design, eliminating the need for bushings and by permitting reduction of weight through reduction of section thicknesses. So far as the work of the foundryman is concerned it assists in giving greater latitude, increasing the density and pressure tightness, strength and

uniformity throughout varying sections and reducing the tendency to chill in thin sections. It also improves the casting properties of the metal and enables chills and denseners to be eliminated without risk of porosity. Uniform machinability and the high degree of finish obtainable are also important features.

The Spectroscope in Metallurgical Analysis

Technique Simplified

THE growing complexity of industrial alloys; the increasing appreciation of the importance of small quantities of impurities; the necessity of a rapid decision in buying ores, spelters, etc., and the ease of spectrographic analysis in certain instances where chemical analysis is almost impossible, are all factors which have combined to make the adoption of the spectrograph in large metallurgical concerns very rapid in the last few years. During the last few years accessories and methods have been perfected which make metallurgical spectrography so truly quantitative that many chemical routine examinations can be relinquished in favour of purely spectrographic ones. With modern instruments the technique is easily learned by any intelligent laboratory assistant, according to the introductory note given in the fifth edition of *The Practice of Spectrum Analysis with Hilger Instruments*, published by Adam Hilger, Ltd., of 24 Rochester Place, Camden Road, N.W.1 (price 3s. 6d. net).

This useful little work is compiled by F. Twyman, F.Inst.P., F.R.S., and the contributors include Dr. S. Judd Lewis, who has for many years used spectrography in his daily work as consulting chemist; Mr. D. M. Smith, the spectroscopic investigator to the British Non-Ferrous Metals Research Association; Mr. S. Barratt, head of the Spectroscopic Laboratory, University College, London; Dr. A. A. Fitch, of the Imperial College of Science and Technology; and Mr. J. W. Ryde, of the General Electric Co. Research Laboratories, Wembley, where spectrum analysis is in regular use. In addition there is a note on emission spectra by Professor E. N. da C. Andrade, of University College, London. A bibliography of nearly one hundred references is included as an appendix.

Carnegie Metallurgical Advisory Board

Annual Meeting at Pittsburgh

THE fifth annual meeting of the metallurgical advisory board to the Carnegie Institute of Technology and the United States Bureau of Mines was held at Carnegie Institute of Technology, Pittsburgh, on Friday, October 16. Progress made on research problems undertaken jointly by Carnegie and the Bureau of Mines was discussed during the morning and afternoon sessions by investigators from the two laboratories. The morning meeting was devoted to reports and discussions on iron-manganese-carbon alloys and chrome-nickel alloys. Reports on research work were given by Dr. Francis M. Walters, Jr., director of the Bureau of Metallurgical Research; Dr. V. N. Krivobok; Maxwell Gensamer and Cyril Wells. Dr. C. H. Herty, Jr., physical chemist, and M. B. Royer, assistant metallurgist, of the Bureau of Mines, gave a report on the solubility of carbon in iron-manganese-silicon alloys. Dr. G. R. Fritterer, associate metallurgist of the Bureau of Mines, reported on the electrolytic method for the determination of inclusions in steel. The physical chemistry of steel making was reported on and discussed at the afternoon session. The three outstanding contributions from this work are the development of a new manganese-silicon deoxidiser, which has been shown to be much superior to ferro-manganese ferro-silicon in combination in producing clean steel at a low cost; the development of a method for quantitatively determining non-metallic inclusions in plain-carbon steels; and the determination of the factors which affect the oxidation of steel in the open-hearth furnace. These reports were made by Dr. Herty and members of the Bureau of Mines staff. Mr. Charles F. Abbott, executive director of the American Institute of Steel Construction, Inc., was the principal speaker at the evening session, his subject being "Market Research in the Steel Industry." Dr. Thomas S. Baker, president of the Carnegie Institute of Technology and organiser of the advisory board, presided at this session.

Metallurgical Topics : Monthly Notes and Comments

From Our Own Correspondents

Simplifying Metallographic Research

SINCE 1912 Professor Dr. M. v. Schwarz, of the Technical High School, Munich, has been trying to develop a method that employs polarised light for the microscopical examination of metal alloys and after much research he has now opened out new fields of employment. The most important result obtained by the employment of polarised light is the fact that on the basis of the phenomena observed, regularly crystallising substances may be separated from all other crystals, this being due to the fact that the former type remain dark when being examined under crossed Nicols, all others being of double refracting character. Rotation of the polarisation plane is the result and owing to the employment of crossed Nicols, the various colours of polarisation clear up or darken four times alternately when turning a metal slide through 360°. In this way clearly perceptible colour changes can be produced which reveal interesting facts about the metal under examination. This discovery is fully discussed by Dr. Schwarz in the October issue of *Metallurgia*, where microphotographic illustrations of non-ferrous alloys are reproduced in the colours obtained as a result of the use of polarised light. The rapidity by which specimens can be examined by this polarisation method offers remarkable possibilities and it may be assumed that this method will, in due course, become one of the indispensable means of scientific research work.

The Practical Application of Research Results

THE increasing recognition of the vast importance, to industry, of the early application of improved methods resulting from scientific research, to manufacturing processes, and of the fact that, in the future it must play even a more important part than it does at present, has led to the establishment of what are called "development" sections, in some of the research associations, designed to act as a liaison between the research laboratory and the works. It devolves on the liaison officer to bring in a practical form which a manufacturer can understand, the latest results of scientific research, construed, so to speak, on a business footing. One of the best examples of this exceedingly useful and practical development is afforded by the British Non Ferrous Metals Research Association, which, several years ago established a "Development Section," and is busy endeavouring, by its means, to instill into the manufacturer some sense of what science can do for him in the way (a) of cheapening his costs; (b) improving his products, and (c) actually enlarging his markets by the creation of new industries. The section referred to is under a very able and exceedingly tactful "development officer," Mr. G. L. Bailey, M.Sc., who has both the scientific spirit, and the requisite business sense, two qualities which, alas, are seldom combined in one individual. By the formation of this section it would appear that Dr. R. S. Hutton, the Director of the Association, forestalled, by several years, the British Association itself, which only last year organised a development branch of its own. This year that branch held its first meeting, as a subsection of the Association, and promoted a discussion on "Bridging the Gap between the Birth of an Idea and its Industrial Application." This is essentially the task of a research association but Dr. Hutton is not content with solving what is, after all, but half of the problem of "getting research results adopted in practice." His method is, after having tried out a given process on a semi-commercial scale in the laboratory, to take it to the works, actually demonstrate its practicability and the advantages, and to stand over the management until they adopt it as part of their ordinary routine. It is to that end that the Development Section of the B.N.F.M.R.A. was founded.

What is "Mild Steel" ?

THE field of research covered by the Institute of Metals seldom overlaps with that of the Iron and Steel Institute, which deals, of course, with alloys in which iron predominates as well, unless that metal is an essential constituent in an otherwise non-ferrous alloy. The subjects of tinning, hot-

galvanising, sherardising and calorising may, however, be regarded as common ground to both Institutes. Thus while tinning problems were discussed at Swansea, "The Attack on Mild Steel in Hot Galvanising" was the subject of a paper presented at the Zürich Meeting of the "The Metals." It dealt with the loss in weight of a ferrous material subjected at varying temperatures and for varying periods, due to contact, in an operation akin to galvanising, with zinc and spelter of varying compositions, and endeavoured to draw therefrom some practical conclusions relative to the "life" of galvanising kettles. The paper was interesting but has little real practical value, seeing that the basis metal was of a nature that hardly occurs in commerce at all, and would certainly not be recognised by iron and steel men as a "typical mild steel," by which terms it is described by the author. It contained 0.054 per cent. C. The term "steel" is apt to be strained; indeed the late Professor Howe used to contend that whatever was made in a steel furnace, or by a process similar to that by which steel was produced, became, *ipso facto*, steel, despite the contrary view held by some of his opponents that being born in a cowhouse would not make a foal a calf. Yet mild steel is a fairly well understood term, and does not correspond in practice with a 0.054 carbon material, which so low a carbon in any ferrous material, so far from being "typical," suggests an overoxidised product hardly suitable for experiments of the nature described in the paper.

Institute of Metals Papers

THE range of the non-ferrous metals being as wide as it is papers read before the Institute of Metals usually cover a fair number of them, but those presented at the Zürich meeting exceed in range, as they exceed in number, those presented at any previous meeting. They fall, as usual, into well defined groups. Thus copper and its alloys constitute the subject of 10 papers, but aluminium and its alloys are dealt with in four only. Others deal with alloys of silver, gold, mercury and magnesium, and others again, on general principles common to all metals and alloys, such as cold-rolling; hardness testing; wire-drawing, and spectroscopic analysis. The latter paper, by Dr. H. W. Brownson and Mr. E. H. S. van Someren is a valuable summary of the present position of this method of research, with notes on precautions necessary in applying it and on the conditions which it is necessary to preserve. The spectroscopic analysis of brass is more specifically described and relative intensity tables are given of the lead, tin, iron, nickel, aluminium and manganese lines met with in alloys of this class, as well as notes and tables on the occurrence of cadmium, arsenic, bismuth, copper, zinc, and silver in lead and its alloys, in which thallium, iron and mercury are likewise often to be detected.

Developments in Nickel Alloys

A BROCHURE describing nickel alloy steels in dredging equipment, in the form of links and pins, tumblers, etc., has been issued by the Bureau of Information on Nickel, which is maintained by the Mond Nickel Co., Ltd., Imperial Chemical House, Millbank, S.W.1. Another brochure deals with nickel-copper alloy condenser tubes in land power stations, the generating stations of the Newcastle-upon-Tyne Electric Co. being described. When these stations were first constructed, the tube materials available were limited to brass and copper, or arsenical copper. These materials did not satisfactorily withstand the prevailing conditions, and as generating machinery was improved, the necessity for better tube materials became more pressing. In many instances brass tubes only gave a life of about one year. As new alloys became available in tube form, their suitability for condenser purposes in the various plants was therefore carefully investigated, with the result that a large amount of data is now available on which to base relative values. Of all the alloys tried, however, those of nickel and copper proved, in the majority of cases, to be the most serviceable. A recent issue of the *Nickel Bulletin* also draws attention to the new nickel alloys which are finding special applications. These are respectively "Hypenik" and a nickel-copper-silicon alloy.

The first named is approximately a 50-50 composition of nickel and iron which has been developed in the United States for electrical applications where moderately high permeability is required together with low hysteresis loss and medium electrical resistance. It has been largely used in radio work, but is perhaps of greater importance in connection with the construction of current transformers and metering instruments. The other alloy is really a Monel metal, silicon being employed to modify the alloy in certain of its applications. The improvements in this alloy have been treated in a paper by Jones, Pfeil and Griffiths, "Nickel-Copper Alloys of High Elastic Limit," presented at the recent Autumn Meeting of the Institute of Metals.

Iron and Steel Trades Opportunities

EXISTING conditions in the iron and steel trades, and the prospect of some measures of safeguarding sufficient at any rate to secure to those trades their own home market should favour a revival of iron ore mining in this country and with it the possibility of reabsorbing some of the unemployed ore miners in the Cleveland and other ore fields. With a depreciated pound foreign ore will cost more, while the economy of keeping workers off the dole is much greater, even if home ore supplies should become a little dearer, than the spread-over cost, to industry generally, of maintaining them in idleness. Where rich ores are required the Cumberland deposits would suffice for many years—certainly long enough to enable the iron and steel industries to turn themselves round and make a fresh bid for their former prosperity. Those ores being non-phosphoric will serve where the highest grade materials are needed while of low grade and phosphoric ores there are available many millions of tons. If scrap becomes dearer, as there is every likelihood that it will, an additional incentive will be added to the more extensive use of pig iron, hence the pig iron trade should share in the revival. Lastly Mr. Vernon Harbord has done useful work in drawing the attention of the trade to the possibility of reviving basic Bessemer steel making in Great Britain. Basic Bessemer steel is suitable, and is indeed used for all the lower grades of steel work employed on the Continent while it is even preferred, in our own country, by many tinplate manufacturers, to mild open hearth steel.

Titanium as a Hardener for Steel

THE potentialities of precipitation hardening, viz., hardening of a character similar to that obtained in Duralumin, are at present engaging the very serious attention of both ferrous and non-ferrous metallurgists. The most recent contribution to the literature (*Kruppsche Monatshefte*, July, 1931, pp. 159-178) deals with the rôle of titanium as a hardener in steels of various types. Experiments made on silicon steels containing varying percentages of titanium indicate that a very marked precipitation hardening could be obtained, but that the increased hardness was associated with a tendency towards coarsening of the grains, brought about by the high quenching temperature. This undesirable feature could be satisfactorily eliminated by suitable additions of nickel, and subsequent experiments showed that the influence of titanium as a precipitation hardener may be exercised to maximum advantage in the case of nickel and nickel-chromium steels; in austenitic nickel-chromium steels containing titanium, it was found possible to obtain hardness values of 611 Brinell hardness number, in conjunction with a fine crystal structure and good tensile properties. In view of conclusions drawn from the earlier experiments as to the importance of silicon as an auxiliary factor in the hardening process, a second section of the investigation was directed to the determination of the influence of other alloy elements on the hardening capacity of titanium; steels of controlled composition were selected for the study of the effect of manganese, nickel, chromium, aluminium and carbon; conclusions drawn relative to the individual elements are detailed. Nickel was found to have a very strikingly beneficial effect, both in enhancing and speeding up the hardening process.

A Novel Electrolytic Process

BUILDING-UP processes by electro deposition of metals, for the repair or reconditioning of worn parts has of course been practised for many years, with considerable success. An interesting innovation in electrolytic methods consists how-

ever of the preliminary removal, by a projected electrolyte, of an unwanted surface, followed by the building-up, by the same means, of another. The process has been applied to the correction of copper plates used in the printing of oceanographic and terrestrial charts, but could, obviously, be applied to many other purposes. In the first place, when removal of a surface is required a stream of copper-plating salt solution is directed to the plate to be acted upon, while the later functions as an anode, the stream itself being in electrical connection from the point at which it emerges, which functions as a cathode. When the surface has been removed to the extent required the operation is reversed, the plate functioning as a cathode, and having a film of easily controllable thickness deposited on it. The necessary corrections can then be engraved on the newly built-up surface. Very high current densities are required in the stream (the electrolyte), up to 3,000 amperes per sq. foot. The voltage varies from 110 D.C. when the plate is the anode, to about 15 when it becomes the cathode. The process is being employed by the U.S. Coast and Geodetic Survey, and was devised by Mr. W. E. Bailey, at the laboratory of the Bureau of Standards, Washington.

Uses for the Rarer Metals

ONE of the features of the modern developments of metallurgy is the extent to which small quantities of the less common metals, either in the pure state or as alloys, are finding employment in various branches of industry. Many of the so-called rarer metals exist in combination in abundance in the earth, but are extracted with difficulty from their ores, when a sufficiently great demand for a particular metal arises, however, it is often found that methods can be developed to produce the metal at a reasonable price—an economic proposition which has proved to be true in the case of aluminium in particular. Metallic barium (99.95 per cent.) has recently been produced as a commercial article and is supplied to manufacturers of ignition equipment in the motor car industry, in which it is used as a high-nickel alloy. In the radio industry the metal is used to clean up the last traces of gas in vacuum tubes. It is expected that the consumption will increase to a substantial amount in the near future. The electrical and radio industries now employ pure molybdenum metal in the form of rod, sheet, and wire. Cerium, when alloyed with iron, zinc, or other base metals, produces pyrophoric alloys, which are used in pocket-lighters. Cerium metal and ferro-cerium are also powerful reducing agents and offer excellent opportunities for industrial research. Both the metal and its alloy can now be produced at a relatively low cost and abundant sources of raw material are available. Cesium and rubidium are used in the manufacture of photoelectric cells. Cesium is also useful for eliminating the last traces of air from vacuum tubes. Recently, metallic lithium has found employment as a de-oxidising agent in the refining of copper; it does not appreciably reduce the electrical conductivity of the copper.

Magnetic Alloy Developments

COMMERCIAL alloys in the magnetic field must have high permeability, negligible hysteresis loss, ductility, and, at the same time, be non-corrosive. The Westinghouse Research Laboratories have been working on such alloys steadily for many years; and Dr. T. D. Yensen has developed an alloy, first made by him in 1923, to the point where it is now ready for wider commercial application. Purity is a very large factor in the preparation of these magnetic alloys. Smallest quantities of disturbing elements alter the regularity of the arrangement of atoms with losses in magnetic properties. Symmetry in molecular arrangements seem to be directly connected with magnetic strength. Studies in crystal structure and space lattice arrangements have been of great value in working out commercial alloys of this type. The perfect magnetic metal would have infinite permeability and zero hysteresis loss. Magnetism, being a corner-stone of the electrical industry is, through research, contributing to industry in new ways. Dr. Yensen first worked on silicon iron, bringing it to a high state of perfection as a magnetic material. Since 1916 he has been with the Westinghouse Laboratories and has developed a method of treating iron nickel alloys in an atmosphere of hydrogen at temperatures from 1,000° to 1,300°, thereby removing small amounts of impurities, chiefly oxygen.

The Reactivity of Coke

Its Influence in Metallurgical Processes

MANY methods have been proposed for measuring the activity of coke in such chemical reactions as oxidation with air, steam or carbon dioxide, terms such as "combustibility" and "reactivity" being used without any precise definition of their meaning. The recent report to the Blast Furnace Committee of the Iron and Steel Industrial Research Council, by J. G. King and J. H. Jones, presented at a meeting of the Institute of Fuel, September 23, however, deals particularly with the "reactivity" of cokes towards carbon dioxide, reactivity being a measure of the velocity of reaction, determined at a temperature of 950°C., under defined arbitrary conditions.

Correlation of Reactivity Value

This investigation has been made for the purpose of explaining why cokes behave differently towards carbon dioxide under similar experimental conditions, as well as to correlate "reactivity values" with the known properties of the cokes in metallurgical practice. It is clear that both the character of the coal used and the conditions of carbonisation exert an influence on the reactivity of the coke, but it has not been possible to make any precise distinction between these two factors. Much more work will be necessary before it is possible to give any general statement on this point; at present it seems that whereas with one coal a change in the method of carbonisation may have a marked influence on the reactivity, with another the effect may be negligible. For instance, in one case, compression of the charge produced a marked change in reactivity, but no change in shatter index, while in two other cases the reactivity values and curves were similar though differences were observed in the shatter indices. It would therefore seem that in assessing differences due to changes in the conditions of carbonisation, the influence of the character of the coal is of primary influence.

The factors which the laboratory investigation indicate to be of importance are (i) the high reactivity of cokes containing reducible iron; (ii) the duration of this high value under the conditions of the process; (iii) the possible production of high reactivity by conditions under which metallic iron constitutes the stable phase; (iv) the effect of the temperature of the process upon reaction velocities; and (v) the area or the character of the carbon surface.

Coke for Blast Furnaces

The investigations recorded have not provided any definite answer as to whether high or low reactivity is most desirable in coke for blast furnace purposes. A high "solution" loss is associated with the former type and possibly a lower rate of reaction at the tuyères with the latter type. The examination of the reactivity of one particular coke as charged and after reaching the tuyères has, however, provided one possibly significant fact. The coke from the tuyère level was in every respect more reactive than that supplied to the furnace; the reactivity curve fell slowly from 165 to 105 after the passage of four litres of carbon dioxide, whilst that of the original coke fell from 140 to the value of about 55 after the passage of only one litre. Further examination of the cokes showed that the increase of reactivity was not due to increase of iron content but entirely to conversion of part of the iron to the reducible state by the action of the furnace gases in a manner analogous to that of controlled oxidation at 450°C. in the laboratory. This observation suggests the possibility of the production of coke of low reactivity which would give a low solution loss but which, after oxidation above the tuyères, would become reactive and increase its efficiency in that zone.

Co-ordination in the Metal Industry

Birmingham Plea for New Technical Centre

THE important part played in industry by the metallurgist was emphasised in the speeches at the sixth annual dinner and dance of the Co-ordinating Committee representing the Staffordshire Iron and Steel Institute, the Birmingham Metallurgical Society and the local section of the Institute of Metals, held at the Queen's Hotel, Birmingham, Saturday, October 31. Mr. W. F. Brazener, chairman of the committee, presided, and the guests included the Lord Mayor of

Birmingham (Alderman W. W. Saunders), Mr. Richard Seligman (president of the Institute of Metals) and Mr. E. W. Bache (president of the Birmingham Chamber of Commerce).

The Lord Mayor, who proposed the toast of "The Co-ordinated Societies," said the city of Birmingham made its reputation as a centre of hardware manufacture, and the greatness of that reputation was not always realised by Birmingham people. Metallurgy was a vital part of the hardware industry that had made Birmingham a great city, and the work of the metallurgical chemist was one of the rocks on which the industry was constituted. In Birmingham, as elsewhere, the hardware industry had suffered a great reverse during recent years, but it was holding its own there as well as anywhere. The metallurgical experts were the men behind the scenes, and their work was of the greatest importance.

The Chairman, who replied to the toast offered two observations, one in the form of a mild grievance, the other a suggestion. His grievance was that frequently industrialists in that and other cities did not make sufficient use of their metallurgists. As the industries of their city were built up mainly on metal products everyone would see that the metallurgist ought to be called in or ought to be on the spot to advise and control the operations in connection with metals. Passing to his suggestion, he referred to a proposed scheme for the establishment in Birmingham of a new technical centre. Here, he thought, it might be desirable to get into touch with the leaders of the technical societies to see if it was not possible in that building to house the technical societies of the city.

Brittleness in Copper

Possibility in Copper free from Arsenic

THAT brittleness in copper is not restricted to "arsenical-copper," but may be produced in copper which is free from arsenic, is the conclusion drawn from a paper by Clement Blozcy, presented at the recent Autumn meeting of the Institute of Metals, which was held in Zürich. The necessary conditions for brittleness, so far discovered, appear to be thorough poling of the copper and the presence of a small quantity of bismuth; present investigations have not yet brought to light any element other than bismuth, which can be regarded as a contributory cause.

Brittleness is developed by cold-working the copper and annealing it below a certain critical temperature which is in the neighbourhood of 650°C., by slowly cooling it from above this critical temperature to any point below, or by quenching from above the critical temperature and reheating just below it. When the copper is annealed above the critical temperature and quenched, it is quite free from brittleness. Metal which has been annealed just below the critical temperature, and quenched, is almost as brittle as metal which has been annealed several hundred degrees below it, but metal which has first been annealed well above the critical temperature, quenched, and then reheated below that temperature, develops brittleness more slowly than does metal which has first been annealed just above that temperature, quenched, and then reheated below it. In a series of alloys made from the one type of copper the critical temperature has also been found to vary according to the bismuth and oxygen contents. In such brittle metal the fracture invariably occurs along grain boundaries. Annealing in air at a high temperature reduces the susceptibility to brittleness to a considerable degree, apparently on account of penetration of oxygen from the surface inwards.

So far brittleness has been produced in commercial arsenical-copper containing phosphorus and appreciable quantities of other elements such as nickel, antimony and lead; high-grade commercial arsenical-copper containing phosphorus and only very small quantities of other elements; and in high-conductivity (wire-bar) copper containing only very small quantities of elements other than copper and oxygen. Experiments carried out to test the influence of selenium proved that the intensity of brittleness was not increased when this element was present in relatively large quantities.

Bethlehem Steel Corporation.

THE directors of the Bethlehem Steel Corporation, New York, have declared a dividend of 50 cents on the common stock.

Some Inventions of the Month

By our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature published weekly in THE CHEMICAL AGE.

Agglomerating Ores

PROCESSES for agglomerating finely divided ferruginous ores, concentrates, etc., in which a thin layer of the material is fed through a chamber and is subjected in admixture with fuel to the action of air under pressure, heated to 650-800° C., are described in Specification 352,832, International Convention date October 30, 1929, by A. Folliet and N. Sainderichin, of Paris. If the ore contains no sulphur it is first mixed with the required amount of fuel and the layer is subjected to the jets of hot air over only a comparatively short length near the point of discharge. If the ore contains sulphur the fuel is added only in the last zone and the air jets are applied to that zone and to an adjacent zone of limited length. For the treatment of materials containing sulphides of arsenic, lead, zinc, etc., a rotary kiln such as that described in Specification 337,636 (see THE CHEMICAL AGE, Vol. 24, Metallurgical Section, p. 18) may be used.

Cast Iron

A GREY cast iron is made by tapping into a ladle a charge consisting mainly of steel, and including some coke or like carbonaceous material, and graphitising the charge in the ladle with an agent which includes a metal. The charge should be of such a composition as would give a white iron on casting and may suitably comprise 60 per cent. of steel, over 12 per cent. of coke, and sufficient silicon-bearing material to give a silicon content of 0.75-1.25 per cent. in the tapped metal, the remainder being scrap, ferro-silicon, nickel alloys, etc. Aluminium or nickel or both may be present in addition to silicon in the graphitiser, with or without deoxidisers and/or other alloying metals. See Specification 352,510, International Convention date January 5, 1929, by International Nickel Co., Inc., of New York, assignees of F. B. Coyle, of Jersey City, New Jersey, U.S.A.

Coating with Metals

METAL articles, particularly of iron or steel, are provided with a protective metal coating, according to Specification 352,202, dated June 10, 1930, by W. H. Cole, of Paris, by heating them in contact with a powdered mixture consisting of cupric oxide, aluminium, chromium oxide, and a very hard abrasive substance such as emery or carborundum. Powdered zinc may also be present and borax, ammonium chloride or naphthalene may be used as a flux. The treatment is preferably effected in a rotary cylinder, hermetically sealed and heated externally, and the heating is preferably in three stages, viz., 30 minutes at about 600° C., 20 to 30 minutes at about 500° C. and 30 minutes or longer at about 350° C.

Iron

To obtain sponge iron the ore is mixed with less than 25 per cent. of its weight of pure carbon and the mixture is heated for several hours in the retorts of a regenerative coke oven, a reducing gas being introduced toward the end of the reduction in order to remove oxygen completely. The sponge iron is then removed in a horizontal direction either by the use of a box-like ladle or by pushing it into a receptacle in which it is protected from atmospheric oxidation. The charge is heated by combustion of gas in adjacent chambers, a reducing temperature of 1,000° C. or higher being specified. The reducing gas may be wholly or partly produced in the retorts. See Specification 353,180 date June 12, 1930, by I. Rennerfelt, of Djursholm, and I. C. S. G. Bohm, of Stockholm, Sweden.

Recovering Metals from Scrap

SCRAP metallic materials, especially scrap copper-molybdenum or copper-tungsten welding electrodes, are ground to powder and heated in a reducing atmosphere. The product may be reconverted into electrodes or other articles with or without the addition of more metal. See Specification 353,377, International Convention date February 4, 1930, by Ford Motor Co., Ltd., London, assignees of R. H. McCarroll and G. Vennerholm, of Dearborn, Michigan, U.S.A.

Refining Metals and Alloys

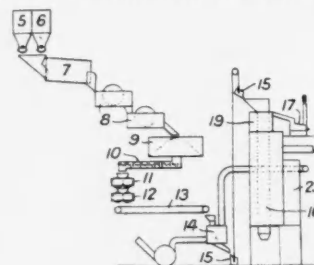
A PROCESS for refining aluminium, magnesium, or alloys thereof by treatment of the molten metal with hygroscopic compounds such as zinc chloride or magnesium chloride is described in Specification 352,199 dated June 5, 1930, by H. C. Hall, of Derby. The compound is introduced in the form of a hard homogeneous mass coated with a waterproof material such as wax, grease, oil, or varnish. Substances specified as suitable for addition to the hygroscopic compound to form the mass are the chlorides of sodium, potassium, lithium, barium or ammonium, and the fluorides of sodium and potassium.

Treating Metallurgical Slimes

METALLURGICAL slimes which are to be blast-roasted are first mixed with solidified or other coarse material, e.g., an agglomerate or a coarse ore, which favours the escape of water, and the mixture is drained, preferably by filtration or centrifuging, and transferred direct to the blast-roasting process. See Specification 353,212, International Convention date June 28, 1929, by Metallges. Akt.-Ges., of Frankfurt-on-Main, Germany.

Zinc

A PROCESS and plant for reducing zinciferous materials by agglomerating a mixture of the ores, etc., and carbonaceous material, and coking and reducing the agglomerates, are described in Specification 353,371, International Convention date December 30, 1929, by New Jersey Zinc Co., of New York. Assignees of E. H. Bunce, of Palmerton, Pennsylvania, U.S.A. The agglomerating, coking and reducing operations are each substantially continuous and are such that no substantial accumulation of material occurs except



353,371

such as is required to maintain a reserve in advance of the agglomerating operation. In the arrangement shown ore and coal from hoppers 5, 6 pass through a mixer 7, and mills 8, in which a binder may be added if necessary, to a bin 9. The mixture passes thence through a paddle mixer 10, in which water is added, to grooved rolls 11 forming small agglomerates which are fed to briquetting presses 12. The briquettes are fed by a conveyor 13, to a coking furnace 14, and the coked briquettes are raised by buckets 15 to a gas-heated reduction retort 16, with an associated recuperator 23, from which the gases pass to the furnace 14. Lead may be removed from the zinc vapour by suitable agglomerates contained in a chamber 19, arranged between the retort 16 and the condenser 17.

Difficulties in the German Metal Industry.

SEVEN hundred iron and metal firms in the Arnsberg area of Westphalia have petitioned the Ministry of Economics to decree a moratorium for old debts. They emphasise that exports have been greatly hampered through the sterling decline. The firms in question deal most with the finished products of the metal industry.

International Nickel Company Staff Changes

CONSEQUENT on the death of J. L. Agnew, vice-president of the International Nickel Co., of Canada, J. C. Nichols, general manager at Copper Cliff, Ont., has been promoted to be assistant to the president at Toronto. Donald MacAskill, manager of the mining and smelting division, Copper Cliff, has been made general manager, and Dr. J. F. Thompson, assistant to the president, becomes vice-president.

Monthly Metallurgical Section

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NOTICE.—Communications relating to editorial matter for our Monthly Metallurgical Section should be addressed to the Editor, THE CHEMICAL AGE, Bowyer House, 154 Fleet Street, London, E.C.4. Communications relating to advertisements and other business should be addressed to the Manager. Contributions will be welcomed from correspondents on any points of interest to metallurgists bearing on works practice or current research problems.

British Research on Non-Ferrous Metals

A Résumé of Recent Progress

The following notes are abstracted from papers which were read before the Institute of Metals on the occasion of their Annual Autumn meeting at Zürich, in September.

AUTOMATIC registering of variation of specific resistance with changing temperature has been applied by J. W. Houghton and R. J. M. Payne ("Transformations in the Gold-Copper Alloys") and by N. S. Kurnaker and N. W. Agnew ("Physics-Chemical Study of the Gold-Copper Solid Solutions") to the study of copper-gold alloys of composition ranging between 20 per cent. and 70 per cent. atomic of gold, and the existence of alloys AuCu and AuCu₃ confirmed during slow cooling. The presence of another transformation in alloys approximating to the concentration of the compound Au₃Cu₂ has also been established.

Experiments have been undertaken to indicate the source and cause of some isolated cases of failure observed in bars of high strength brass. It has been determined that the reeling process for straightening, after extrusion, in the case of a brass with a high yield-point, is responsible for residual stresses of such an order that failure due to internal cracking may result during storage, or on subsequent machining. Suitable low temperature annealing treatment effectively removes all possibility of residual stress cracking. It has also been found by D. G. Jones, L. B. Pfeil, and W. T. Griffiths ("Nickel-Copper Alloys of High Elastic Limit") that the elastic limit is low in substantially pure nickel-copper alloys in the fully annealed and in the cold drawn conditions, but that high elastic limits are developed in all compositions as a result of low-temperature heat-treatment following cold-working. High elastic limits may also be produced in nickel-copper alloys containing small amounts of such elements as silicon, which render the alloys susceptible of heat-treatment.

Copper-Zinc Alloys

The behaviour, under oxidising conditions, of copper-zinc alloys containing from 60 to 100 per cent. copper has been examined by S. Dunn ("The Oxidation of Some Copper Alloys") with respect to time, temperature and composition of the alloy. Chlorine compounds have been found to accelerate the rate of oxidation. Nickel and arsenic in small quantities have been found to be without influence, but aluminium, even when present to the extent of 0.1 per cent., has been shown to exert a marked protective action. While silicon confers some measure of protection in alloys studied with a silicon content up to 4.5 per cent. at 725° C., at 827° C. this effect breaks down.

In the physical testing of copper and copper-rich alloys in the form of a thin strip, consistent results are obtainable for tensile strength and elongation on all materials down to and including 0.02 in. thickness. One of the most—if not the most—satisfactory methods of measuring hardness according to M. Cooke and E. C. Larke ("Physical Testing of Copper and Copper-Rich Alloys") is found to be the diamond pyramid static indentation method. Brittleness has been found not to be restricted to arsenical copper, but may be produced in copper free from arsenic. The necessary conditions so far discovered according to C. Blazey in his paper on brittle in copper, appear to be thorough poling of the copper and presence of a small quantity of bismuth. Investigation has not yet brought to light any elements, other than bismuth, which can be regarded as a contributory cause of brittleness.

Aluminium and Aluminium Alloys

The macro-structure of aluminium-silicon alloys can be revealed satisfactorily by means of a solution of cupric

chloride containing from 150 to 160 grm. per litre, in accordance with directions given by W. Hume-Rothery. Etching is carried out by immersing the specimen several times in the copper chloride solution, the deposit of copper being removed between each immersion. A final brightening can be obtained by treatment with a dilute solution of chromic acid.

The presence of water vapour in contact with molten aluminium or its alloys leads to the production of "pinholes" in sand castings of the metal. The most generally successful method of treating the molten metal prior to casting, with a view to eliminating such pin holes, according to papers by D. Hanson and I. G. Slater ("Unsoundness in Aluminium Castings") has been found to be with a mixture of nitrogen and chlorine, by means of which all alloys examined can be made practically sound by a treatment of 20 minutes duration at 700° C. Pinholes also result when virgin aluminium is exposed to suitable corrosive conditions and subsequently sand-cast. The suggestion is made that the deterioration is the result of electrolytic action or corrosion, involving the liberation of hydrogen in the nascent state which is absorbed by the metal. On remelting and casting, the hydrogen is evolved in the molecular condition and produces pinholes.

The Brinell Test

Owing to the shape of the lip of indentations made by the Brinell test in certain cold worked metals, diameter measurements of the indent may differ 3-4 per cent. according to whether vertical or oblique illumination is used with the measuring microscope. A paper by H. O'Neill ("Diameter of Certain Brinell Indentations in Cold-Rolled Metal") therefore suggests that when a Brinell indentation can be made to display more than one "diameter" under the measuring microscope, the minimum reading should be recorded. The effects of cold rolling, heat-treatment, and storage on the Brinell hardness of 14 lead alloys containing small amounts of tin, cadmium and antimony have been studied by H. Waterhouse and R. Willows in their paper on the effects of cold-rolling and of heat treatment on some lead alloys. The hardness numbers of the cast alloys ranged from 5 to 18 Brinell. Cold rolling hardened the soft alloys and softened the hard alloys, the hardness immediately after cold-rolling lying in all cases between 8 and 11 Brinell. Self annealing at atmospheric temperature further reduced the hardness to about 7 Brinell.

Protecting Magnesium against Corrosion

Of the methods examined by H. Sutton and L. F. Le Brocq for protecting magnesium alloys against corrosion, the most promising appeared to be that of a chemical treatment of the surface, followed by the application of lanoline or a suitable enamel. Two chemical processes have been developed which afford very fair protection, viz., (1) immersion for six hours in a bath containing 1½ per cent. potassium dichromate, 1 per cent. alum and ½ per cent. caustic soda, the bath being maintained at 95° C.; (2) immersion under the same conditions in a bath containing 1½ per cent. potassium dichromate and 1½ per cent. sodium sulphate (decahydrate). For surfaces which are in the "as cast" condition, a preliminary cleaning treatment in 10 per cent. nitric acid is necessary, but for parts machined to fine tolerances this method is too drastic. In such cases a solution of 2 per cent. caustic soda is used. Sand blasting as a preliminary treatment is not recommended.

Spectrographic Assay

It has been shown by D. M. Smith ("The Spectrographic Assay of Some Alloys of Lead") that by the application of the spectrograph to the quantitative determination of low percentages of tin, antimony and cadmium in lead alloys, the constituents can be determined within the range 0.1-1 per cent. with an accuracy of 10 per cent. This work has been carried out on behalf of the British Non-Ferrous Metals Research Association, with the object of finding a rapid and reliable method of assay to cover the wide and increasing use of the tenary alloys of lead. The work refers to alloys containing up to 1 per cent. of antimony and cadmium, and up to 3 per cent. of tin, present individually or collectively. Spark spectra give more consistent results than arc spectra; only half an hour is required compared with 5 hours for the chemical assay; and by the ordinary method of direct comparison an accuracy of 10 per cent. within a range of 0.1-1.0 per cent. for each of the alloying constituents can be obtained. This, of course, is a distinct saving of time.

Silver-Mercury Alloys

The constitution of the alloys of silver and mercury has been determined over a range of temperature extending from the melting point of silver to -50°C . Progressive additions of mercury to silver according to A. J. Murphy, in his paper on "The Constitution of Alloys of Silver and Mercury" cause a continual reduction in the temperature of the initial freezing point down to -38.8°C ., the freezing point of mercury. No alloy in the series has a freezing point lower than that of pure mercury. Silver can retain in solid solution 55 per cent. by weight of mercury at 276°C ., the amount probably increasing somewhat at lower temperatures. Two intermediate phases of restricted composition are formed. The beta-phase contains 40 per cent. of silver and dissociates on heating at 276°C . into alpha- and liquid. This phase has a close packed hexagonal lattice in which $a=2.98\text{ \AA}$ and axial ratio $c=1.62$. The gamma phase contains 29-30 per cent. of silver, and dissociates on heating at 127°C . into beta- and liquid; it has a body-centred cubic lattice with side 10.0 \AA .

Melting Nickel-Chromium Alloys

In studying the phenomena connected with the melting of nickel-chromium alloys in hydrogen, C. J. Smithells, S. V. Williams and E. J. Grimwood ("Melting Nickel-Chromium Alloys in Hydrogen") have shown that sound ingots can be produced, provided that all the oxides present in the melt are reduced by hydrogen before casting. If any oxides remain in the melt, these are reduced by the hydrogen, and steam is liberated at the moment of solidification with the formation of blow-holes. For casting in air it is necessary to displace the hydrogen with nitrogen before pouring. The effect of using various grades of raw materials in the preparation of the alloys has been studied. Alloys prepared from commercial materials have been found to give better resistance to oxidation than those prepared from raw materials. The probability that the presence of certain impurities has a beneficial effect was confirmed by making deliberate additions of elements known to be present as impurities in the raw materials. The process has also been used with success for the preparation of other metals and alloys in the ductile state without the use of the usual deoxidisers.

Steel Firm's Surplus Capital

A CIRCULAR to shareholders of Samuel Osborn and Co., of Clyde Steel Works, Sheffield, which has an issued capital of just over £550,000, states that the company is in a very strong position with its gilt-edged and other investments and without a Debenture of charge on any of its assets, and has for some years past had more liquid resources than are necessary for normal trading purposes. The directors, however, have been contemplating the possibility of using part of these liquid resources to pay off some of the existing capital, but are advised that instead of reducing the capital, the simplest and the best method of dealing with the matter is to wind up the existing company and form a new one with the same name, the new company to acquire the assets of the old company.

Research on Nickel Steel**The Influence on Silicon**

THE properties of nickel-silicon steel, covering a wide range of compositions, were described by Guillet in 1906. (*Journal Iron and Steel Institute* 1906, No. 2). Although the exact limits of composition of the structural diagram were not determined, it is clear from his work that all medium-carbon steels containing up to 4 or 5 per cent. of nickel and 3 or 4 per cent. of silicon are essentially similar in structural constitution, and fall well within the pearlitic group, though with the higher silicon there was a slight tendency for the formation of martensite at the rate of slow cooling he employed. Guillet found that the presence of silicon raises the transformation points of nickel steel, and that silicon raises the elastic limit, tensile strength and hardness of nickel steels, but only at the expense of the elongation and the impact figure, especially at right angles to the plane of rolling. His final conclusion was that the addition of silicon is not to be recommended.

A recent investigation by R. Harrison, of the Research Department, Woolwich, connected with the Iron and Steel Institute at their Swansea meeting (September 29-October 2) deals with nickel steels suitable for various constructional purposes, in which it is customary to limit the amount of silicon to 0.3 per cent. Its object was to determine the effect on nickel steel of silicon up to about 1 per cent., and, if the results were of sufficient promise, to extend the work to steels of higher silicon content. There is no reason to anticipate a sudden change in the character of the effect of silicon, since the structural constitution remains the same up to at least 3 per cent. of silicon. The second part of the investigation was not proceeded with, however, since the work completed revealed no probable advantage to be expected from the increased silicon content. The steels used in this investigation were crucible steels, cast into ingots about 4 in. square, and rolled into bars $1\frac{1}{2}$ by $\frac{3}{4}$ in. in section. They contained 4 per cent. of nickel and about 0.4 per cent. of carbon. The silicon ranged from 0.05 to 0.87 per cent. in two series having different manganese contents, namely, 0.4 and 0.75 per cent.

Brinell hardness tests were made on small pieces of the steels, water-quenched from 850°C . and tempered at various temperatures between 200°C . and 630°C . In all cases the same typical form of tempering curve was produced, the hardness after any given treatment being displaced in an upward direction by increasing carbon, increasing manganese or increasing silicon.

Effect on Critical Points

The chief conclusion drawn from this investigation was that in nickel steels containing 4 per cent. of nickel, 0.4 per cent. of carbon, 0.3 to 0.8 per cent. of manganese and 0.05 to 0.87 per cent. of silicon, silicon raises the critical points, both on heating and cooling. The effect of silicon on 4 per cent. nickel steel, whether in the forged, rolled, or normalised condition, is a hardening effect. It increases the elastic limit, yield point and tensile strength, but reduces the elongation, reduction of area and notched-bar impact figure. For use as plates in the rolled or normalised condition, irregularity of properties due to partial air-hardening must be guarded against by keeping the silicon, as well as the manganese, below 0.5 per cent. A similar hardening effect due to silicon is evident in steels which have received the same hardening and tempering treatment; but in small sections, hardened and tempered to give a stated hardness, the silicon has very little effect on the properties. As the size of the section is increased, mass effect begins to show first in steels lowest in silicon. When the manganese is insufficient to ensure full hardening, the effect of a slight increase in manganese is greater than that of a considerable addition of silicon. When the manganese is already at a high figure, an increase of silicon maintains a hardened condition in somewhat larger sections. The usual limit for silicon is 0.3 per cent. Provided that homogeneous steels free from laminations are secured there is no objection, from the point of view of mechanical properties, to the use of a considerably higher silicon content, except in rolled nickel-steel plate, in which it should not exceed 0.5 per cent. On the other hand, no advantage is gained by abnormally high silicon. The steels investigated contained not more than 0.87 per cent. of silicon, but they gave no indication than any specially good mechanical properties are obtained by increasing the silicon to 1 or 2 per cent.

Metallurgical Topics : Monthly Notes and Comments

From Our Own Correspondents

Supplies of Tungsten

PRIOR to the war tungsten ores were chiefly obtained in the United States, Burma, Australia, and to a minor extent in Europe. Like other steel alloy metals, such as molybdenum, nickel, chromium, the demand increased enormously during the war. As a result of the discovery of considerable deposits in certain provinces of China, that country became the centre of production. At the present time the province of Kiangsi with a yearly output of about 5,000 tons is the chief source, but owing to internal strife, mining operations are arrested for months on end with the result that the output is of an extremely fluctuating nature. The considerable fluctuation in the world's production of tungsten is, therefore, not so much due to the question of consumption as to the prevailing political situation. Antimony, for example, is effected in the same manner, China, in this case, too, being the chief producer. The falling off in production has been accompanied by a considerable drop in the quality. Though Bolivian and Spanish production has increased, there has been a falling off in the Portuguese output, and the production of tungsten in America has acquired considerable importance in spite of the cost of mining the ore. The chief consumers of wolframite are the United States, Germany, France, Great Britain, Austria and Italy. The tungsten exports of China, Burma and the U.S.A. in 1928 were 7,246, 612 and 1,079 tons respectively. The production of the Malay States, Spain, Indo-China and Australia varies between 200 to 300 tons annually.

Tin in the United States

TIN is the only metal of which the United States annually consumes more than \$20,000,000 worth and yet remains a negligible producer, according to a recent report by the United States Bureau of Mines. In fact, the value of the tin annually imported for consumption ranges from \$60,000,000 to over \$100,000,000 and accounts for almost half of the world production. Annual domestic production is valued at less than \$50,000 and amounts to less than one-twentieth of 1 per cent. of the world's total output. The principal tin-consuming industries are food packing, automobile manufacture and building. The packing of food is relatively stable from year to year and may be counted upon to absorb a fairly constant quantity of tin plate and solder, but the automobile industry is subject to wide variation in the number of units produced. If the recovery of secondary tin—that is, the production of tin from sources other than ore—were included with primary production, the United States would rank third among the tin-producing countries of the world. Tin-bearing alloys, tin-plate clippings, and melting pot drosses are the most important materials from which tin is reclaimed. Most of the tin recovered from alloys does not pass through a refined tin-stage but is made into alloys which are brought to the required specifications by the addition of virgin metals. Tin plate entering the reclaiming processes consists chiefly of trimmings incidental to the fabrication of tin-plate products. In the past some used tin cans have entered the detinning plants but, at present prices for reclaimer metal, the recovery of tin from this source is unprofitable in the United States.

Electronic Emissivity of Metals

SIR HAROLD CARPENTER has recently expressed the view that the metallurgy of the future will have to be more closely allied with physics than it has hitherto been, while Dr. Rosenhain has often shown that physical rather than purely chemical considerations loom largely in most metallurgical problems. The electronic state of metals is attracting more and more interest and attention and amongst many valuable contributions to this subject, that by Dr. Karl T. Compton, of the Massachusetts Institute of Technology, on "Emission of Electrons from Metals" recently appeared in the *Journal of the Western Society of Engineers*. Such emission may be said to be intimately associated with many of the most modern inventions and discoveries, and likely to lead to others, the outcome of which can hardly, even yet, be foreseen. In the

meanwhile the study of such phenomena throws much new light on the nature of metals, and serves too, to reveal the presence of impurities not to be detected by any other means.

Some Applications of the Phenomena

Electronic emission can be induced by five different methods: (1) by heating a metal to a very high temperature (thermionic emission) (2) by illumination of metal by suitable forms of radiation (the photo-electric effect) (3) by bombardment (4) by contact between metals at enormous differences of potential, and (5) by applying enormous potentials. Some of these methods can be applied simultaneously. In thermionic emission there is some analogy with ordinary evaporation from a surface, while, similarly, the rate of emission can be controlled by the voltage applied to the emitting metal, just as evaporation can be controlled by controlling the temperature at which it takes place. Work on the photo-electric effect has enabled the fundamental constant of the quantum theory to be accurately ascertained (Planck's constant). The more mechanical modes, impact, contact, etc., have also thrown light on many problems. From the prosaically practical point of view it may be said that new forms of lighting, new methods of analysis and new developments in television and transmission wait on further researches on the theory and applications of electronic emissions from metals.

Nickel-Chromium-Iron Alloys

EXTENSIVE investigations dealing with a theoretical study of the chromium-nickel-iron-carbon diagram, with factors influencing the existence of metastable phases, and with "decomposition" and "disintegration" phenomena characteristic of these alloys have recently been reported by V. N. Krivobok, jointly with other investigators, before the American Society for Steel Treating. It is suggested that the constituent precipitated during the disintegration is not necessarily a carbide; in very low-carbon alloys, it is undoubtedly magnetic chromium-nickel ferrite, indicating that the nature of the constituent separated is dependent on the carbon content. A phase change, from non-magnetic to magnetic, accompanies decomposition. A range of austenitic alloys of varying carbon content was studied by means of magnetic analysis, microscopical examination, and corrosion tests in copper sulphate-sulphuric acid, and dilute acid solutions. Alloys of extremely low carbon content, in which it is impossible for carbide precipitation to occur, are shown to be free from susceptibility to disintegration, but when the carbon is increased above a very low percentage, the decomposition process is practically independent of carbon content. The authors therefore conclude that mere lowering of the carbon to the values commercially obtainable does not render the alloys immune from decomposition. A study of the influence of additions of cobalt, molybdenum, silicon and copper in inhibiting disintegration, showed that certain of these elements prevent phase change but do not suppress precipitation of carbides; their effect appears to be materially influenced by the percentage of carbon present. Alteration in the ratio of nickel to chromium was not found to decrease susceptibility to disintegration. Correlation of the results of tests in copper sulphate-sulphuric acid, with other acid-corrosion tests and with the mechanical properties of the alloys, indicates that the "standard" weld decay test should be accepted with caution as an exhaustive criterion of susceptibility to corrosion disintegration and liability to mechanical weakness.

Some Foreign Metallurgical Researches

THE Intelligence Department of the British Aluminium Co. deserve the thanks of all metallurgists fortunate enough to be favoured with their two excellent monthly issues *Broadcast*, and *Light Metals Research*, which do for the industries in which the company is interested what that equally excellent publication *The Nickel Bulletin* does for the products of the Mond Nickel Co., Ltd. In the current number of *Light Metals Research*, the Editor, Mr. Freeman Horn, has rendered available to those concerned with the problem of corrosion, a very full translation of a very valuable paper on "The

Investigations of the Corrosion of Light Alloys," from the October issue of the *Bureau International de l'Aluminium*. This investigation embodies the team work of a number of investigators, and lays down principles to be followed in corrosion research if concordant and comparable results are to be obtained. It emphasises the necessity of stating accurately the analysis of all samples subjected to corrosion tests, as well as the heat treatment, and mechanical treatment they may have received. It recommends the use of test pieces of uniform dimensions; careful observance of temperatures, the necessity of proper methods of suspending test pieces, and of proper methods of cleaning them. Finally it gives detailed descriptions of all the best known methods of testing corrosion resistance, and the various modes of carrying out field and accelerated tests. The same issue contains an equally valuable translation, from the German, of a paper on "The influence of small quantities of impurities in metals," by Dr. Fraenkel, read at a recent meeting of the *Deutsche Gesellschaft für Metallkunde*. This paper has useful tables showing the extent to which purification has already been attained in various metals, and the range within which the spectroscopic detection of minute amounts of impurities can be achieved.

Pig Iron and the Scrap Question

THE implications of the present economic situation and of impending changes in our fiscal system have a very immediate bearing on the course of technical developments in the iron and steel trades. With protection, and a tacit pledge that customers requiring iron and steel as the raw materials of their own industries, iron and steel manufacturers have a stronger incentive than ever to reduce costs and improve practice. It is obvious that iron ore from abroad, used to the extent, at home, of many millions of tons yearly in British blastfurnaces, will cost far more than it has done hitherto—even in the case of British firms owning their own foreign ore mines. Allowing for the depreciation of the pound and an unavoidable increase in freights, such ores can hardly fail to cost from 5s. to 6s. per ton more, and therefore add practically double that to the cost of pig iron. This should stimulate the use of home ores, and although, being leaner, they will need more coke per ton for smelting, the coal trade, as well as the iron ore mining industry should benefit, while improved coking practice and a larger yield of blastfurnace gases should cheapen power fuels all round, and lead, too, to increased by-product recovery and reduced costs in such processes. At present the blastfurnace industry is one of the most depressed of all our home industries. Anything that helped it to recover the ground lost of recent years should have widespread and beneficial repercussions on a number of other industries. At present it is being sadly hampered by the availability of large amounts of scrap, at prices with which pig iron cannot at present compete. Opinions may differ as to whether the best steel is made from steel rather than from pig iron, but the balance is in favour of the quality of steel—and, for the matter of that, of cast iron as well, made from pig iron, rather than from scrap. Besides, is scrap really as cheap as it appears to be? Much of it costs as much to make as steel itself, although of course, really "second-hand" scrap, as distinct from process scrap, is, for the moment, lower priced than pig iron.

Faraday's Alloy Steels

NOT the least of the contributions to the centenary celebrations of Faraday's discovery of magnetic induction is the handsome volume which Sir Robert Hadfield has just dedicated to an aspect of Faraday's work which has not hitherto attracted as much attention as it deserves. This new book (*Faraday and his Metallurgical Researches*; Chapman and Hall, 21s. net) exemplifies how greatly Faraday was in advance of his time and amplifies delightfully the fascinating account Sir Robert has already given of the finding at the Royal Institution, of the famous box containing a collection of alloys of iron with nickel, chromium, copper, silver, and the metals of the platinum group. That earlier account was embodied in a paper before the Royal Society, published in September last, and described the nature, chemical composition, micrographic structure and physical properties of these hundred-year-old specimens. Since then nine further specimens, deposited at the Natural History Museum, South Ken-

sington, have come to light, together with a razor of "wootz" steel, made for Faraday from steel prepared by methods he devised at the Royal Institution. These have been subjected to the same painstaking examination, at the Hadfield Research Laboratories in Sheffield, and the results are given in Sir Roberts' new book. The later specimens comprise alloys of iron, or rather steel, containing up to 48.6 per cent. of platinum, and, in another instance, up to 48.8 per cent. of rhodium. The identification and subsequent investigation of these specimens reveals yet another scientific achievement, seeing how small, in the first place, was the amount of material available, and how essential it was to use as little as possible for the purpose of examination.

Researches on Minute Amounts of Material

THE original box contained 79 specimens weighing, in the aggregate, no more than 7 lb. 14 oz. They were made the subject of 634 separate tests, physical, chemical, and micrographic. Yet so meticulously was the need for economising material of such great historical and scientific value that no more than 1 lb. 3¼ oz. was used up in carrying out the researches. From this inconsiderable amount 357 distinct analyses were made. Faraday possessed, as is well known nowadays, marvellous skill as a manipulator, yet the skill involved in the work done by Sir Robert Hadfield and his staff displays a degree of manipulative skill which has probably never been surpassed. Metallurgists may wonder how the work was done, and may even be disposed to challenge the accuracy of the analytical work carried out on such small quantities of material as well employed. Much work of this nature is published from time to time without any evidence being afforded of the means employed for establishing the results. In the present instance Sir Robert has met every possible objection that could be advanced on that score by giving the very fullest details of the mode of conducting the analyses, and of the methods employed. The tensile tests made were carried out by means of the ingenious Hounsfield tensometer, which enables tiny test pieces to be accurately tested. The preliminary sorting—for the specimens had first to be identified, and later correlated with entries in Faraday's own diaries and note books—was effected, in part, by spectroscopic analysis. The wealth of metallurgical information in Sir Robert's new book endows it with special value to all metallurgical investigators, since it is less an account of what Faraday did, than of what can be done, to-day, in the way of scientific investigation. Apart from this the book possesses the very greatest historical interest as it traces the origin and development of ferrous alloys to which its author has made noteworthy contributions.

Metallurgical Fuel Problems

THE circumstances in which our country now finds itself should be intensely stimulative of new developments in metallurgical technology. Of late what may be called the line of least resistance in power generation has seemed to be in the direction of the ever-increasing use of internal combustion. The very evolution of automobile engineering has been possible only because light fuel oils have been cheap and plentiful while the Diesel engine, which is making a steady bid for the position of the prime mover, *par excellence*, has likewise reached its present state of perfection because the heavy fuel oils have been similarly cheap and widely available. Now, except for the fuel oils, we can ourselves produce at home, practically as by-products of the use of coal for other purposes, all the oil needed will have to be obtained, as hitherto, from abroad, and, with our depleted pound, will cost us far more than hitherto. The only extensive and natural source of fuel we possess is coal, and steam engineering is like to come into its own again but in an enormously improved and much more economic form. Extremely high pressure and temperatures will be the order of the day. The extent to which steam engineering can be rendered efficient enough for the conditions now imposed on British industry will depend upon the extent to which the metallurgist can develop alloy metals capable of withstanding the pressures and temperatures called for. Corrosion resistance at high temperatures is similarly called for. Nickel, and nickel chromium steels, with or without, but probably with, molybdenum, and the important rival group of non-ferrous will then be more in demand than ever.

Chromium Plating

Investigations in Porosity of Deposits

RECENT research by the United States Bureau of Standards in co-operation with the American Electroplaters' Society, dealing with methods for detecting and evaluating the porosity of the chromium deposits were investigated, and the factors that influence porosity were studied, is published in the October issue of the Bureau of Standards *Journal of Research*.

Various methods that have been described for detecting porosity in chromium deposits were found to yield consistent results. The most generally applicable and reliable is the copper deposition method, which depends on the fact that under certain conditions copper will not deposit on chromium, but only in pores or cracks in the chromium. This method was made semi-quantitative by measuring either the average apparent current density or the weight of copper deposited in two minutes at 0.2 volt. The results represent the relative porosities. In order to determine whether a decrease in porosity of the chromium will result in an increased protective value, exposure tests are being made in co-operation with the American Electroplaters' Society and the American Society for Testing Materials.

Very thin deposits of chromium usually contain round pores. As the thickness is increased, these pores disappear but cracks are formed, especially on standing. For the common conditions of deposition, the minimum porosity is obtained with a thickness of about 0.00002 inch, which is the usual thickness applied to automobile parts. If, however, the chromium is deposited at a higher temperature such as 65° C. instead of the usual temperature of 45° C., and at a suitable current density, the deposits are less porous than usual. This is especially true of thicker deposits. The least porous deposits are produced on nickel, which is generally applied as a preliminary coating on other metals before chromium plating. Heating the deposits to 200° C. for a short time produces a greater porosity than if they were allowed to stand at ordinary temperature. Under the latter conditions the porosity gradually increases for two weeks.

Birmingham Iron Trade

Market Preoccupied with Tariffs

ACCORDING to *The Birmingham Post* conditions in the iron and steel trade are opposed to the circulation of business. Eyes are fixed expectantly on Westminster, where the interests of the iron industry as a vital part of our national economy are receiving a great deal of attention. The market will be disappointed if a duty is not imposed on dispensable imports of iron and steel before the adjournment of Parliament. Whether the expected duty will be confined to finished material, or whether it will have wider scope, is a question which is anxiously canvassed. There are, of course, differences of view as to what is expedient in the circumstances. But that iron and steel should be overlooked in the new orientation of public opinion to the problems raised by the balancing of our trade is not credible to anybody.

In these circumstances it is pointed out that neither merchants nor consumers are prepared to accept the risks of buying foreign material. It may conceivably cost them a good deal more than British material, and the only object of buying abroad is to save money. Considerable tonnages are still being shipped against old contracts. Consequently, although there is increased activity in the re-rolling trades, no real stringency has developed in supplies of raw materials. So far as heavy steel is concerned, consumptive demand is backward. There is a partial reinvigoration at the light foundries, hence a sounder equilibrium is being restored in the pig-iron department, as is evident from the rekindling of a certain number of furnaces since the autumn began.

The Central Pig-Iron Producers' Association held its usual monthly meeting at Birmingham on Thursday, November 26, under the presidency of Sir Francis Joseph, and decided to make no change in prices. Coke is advancing; it is now quoted round 12s., whereas it could be bought at about 10s. when pig-iron prices were put on the basis now applying. Most of the furnacemen are covered by contracts entered into when the low prices prevailed; those who have to go on the market at present rates are becoming uneasy about the relation of their costs to their sales returns for pig-iron.

Our Abnormal Steel Imports

Facts and Figures Criticised

COMMENTING on the question raised by Mr. C. Peat (member for Darlington), in the House of Commons on November 24, as to the proportion which imported iron and steel bore to the total home production, Sir William Larke (director of the National Federation of Iron and Steel Manufacturers) in a letter to *The Birmingham Post*, is of the opinion that the reply of the President of the Board of Trade suggests that the imports of iron and steel were negligible in relation to production, an impression which should be corrected. The extremely low figures obtained by the Board of Trade were apparently the result of relating the imports of ingots and castings, in which there is very little import trade, to the production of steel ingots and castings; and ignoring the greater imports of steel in semi-finished forms subsequent to the ingot stage. In 1929, the output of steel ingots and castings amounted to 9,636,000 tons, a monthly rate of 803,000 tons. Since 1929, the production of steel has been steadily declining until, in October this year—the latest month for which figures are available—the output of steel ingots and castings amounted to no more than 457,000 tons. In 1929, imports of steel in various forms amounted to 2,431,000 tons, equivalent to 3,000,000 tons of ingots. Imports of steel therefore represented 31 per cent. of home production. In October, when only 457,000 tons of steel were produced, imports of steel in various forms amounted to 233,900 tons—equivalent to 278,500 tons of ingots; so that in that month imports represented 61 per cent. of production, or nearly twice the rate obtaining in 1929.

Lord Daryngton, in a further letter, which has appeared on this subject points out that if Sir William Larke's figures are not challenged, the necessity for immediate action is strengthened by the fact that foreign nations are either increasing the taxation on coal imports from us, or limiting them, or both; that the recent Coal Act has increased the cost of production by preventing the most efficient collieries producing coal cheaply, by limiting output; and that wage levels in the coal trade cannot be altered under ordinary circumstances before next summer, as they are statutory.

Iron and Steel Industry in Canada

Statistics for 1930

ACCORDING to the Dominion Bureau of Statistics at Ottawa, firms operating plants associated with the primary iron and steel industry in Canada had an output during 1930 valued at \$52,588,935. This value, which represents the amount actually received by producers for the sale of pig iron, ferro-alloys, steel ingots, direct steel castings and rolled iron and steel products, was 27 per cent. below the corresponding figure for 1929 and 15 per cent. below 1928, but exceeded the 1927 value by 15 per cent. The totals amounted to \$72,231,995 in 1929, to \$62,071,674 in 1928, and \$45,571,264 in 1927. The 34 firms in question operated 49 different plants or departments including blast furnaces, steel furnaces, ferro-alloy plants and rolling mills. Capital employed by these concerns was reported at \$112,079,926; the average number of employees was 9,723, and salaries and wages totalled \$14,934,325. Pig iron was made in 4 plants, ferro-alloys in 3, steel in 27, and rolled products in 16 plants.

During 1930 the 4 manufacturers of pig iron in Canada produced 747,178 long tons of pig iron as compared with 1,080,160 tons in 1929, 1,037,727 tons in 1928, and 709,697 tons in 1927. Furnace charges for 1930 included 1,328,929 long tons of imported iron ore, 94,766 long tons of mill cinder, etc., 35,909 long tons of scrap, 401,688 short tons of limestone, and 796,040 short tons of coke. Employees in the iron blast furnace departments averaged 769 per month in 1930. Imports of pig iron into Canada during 1930 amounted to 13,643 long tons, a decline over 58 per cent. from the total of 32,548 tons brought in during 1929. Exports were reported at 593 long tons as compared with 7,478 tons in the previous year. The production of ferro-alloys in Canada during 1930 amounted to 65,223 long tons; production of steel ingots and direct steel castings in Canada totalled 1,009,578 long tons, a decline of 27 per cent. from the 1,378,024 reported for the previous year.

Some Inventions of the Month

By our Patents Correspondent

Abstracts of other Patents of metallurgical interest will be found in our Patent Literature published weekly in THE CHEMICAL AGE.

Alloys

A HARD metal composition suitable for tool bits is obtained by sintering a mixture of powdered tungsten carbide and powdered uranium. Thus the mixture may be heated in a carbon mould at 1,500-1,600° C. A product containing 82 per cent. of uranium, 5 per cent. of carbon, and 13 per cent. of uranium is specified. See Specification 354,028, International Convention date July 10, 1929, by British Thomson-Houston Co., Ltd., of London, assignees of S. L. Hoyt, of Schenectady, New York, U.S.A.

AN alloy steel, resistant to rusting and corrosion, contains 0.1-1 per cent. of chromium, 0.25-0.5 per cent. of copper, 0.04-0.25 per cent. of nickel, and 0.01-0.05 per cent. of tungsten and is free from silicon and poor in carbon. The steel is produced by first smelting in a Siemens-Martin furnace a charge comprising Martin pig iron, a special pig iron containing up to 0.1 per cent. only of phosphorus and scrap iron with the usual addition of lime, and bringing down the carbon content to 0.03 per cent. by decarburising additions of iron oxide, e.g., slag or scale in quantities of 0.4-0.5 per cent. of the charge. After degassing, e.g., by additions of ferromanganese or aluminium the charge is alloyed by adding a ledeburitic carbide graphite-free alloy containing approximately 2.5-3 per cent. of carbon, 20-30 per cent. of chromium, 6-10 per cent. of nickel, 10-15 per cent. of copper, 5-6 per cent. of tungsten and the remainder iron free from manganese, silicon or phosphorus. See Specification 354,177, International Convention date March 30, 1929, by R. Kesselring and J. Nowak, of Oderberg, Czechoslovakia.

ALLOYS highly resistant to corrosion and particularly suitable for dental purposes and as conductors of heat and electricity substantially consist of about 25-98 per cent. of palladium, about 1-50 per cent. of copper and not less than 1 per cent. of silver. The presence of iron and similar metals apt to impair the ductility is to be avoided. When applied to castings a deoxidiser such as manganese, zinc, silicon, boron, magnesium or calcium or compounds thereof, e.g., 0.2 per cent. of calcium boride, may be present. The preferred heat treatment of the alloy comprises heating to above 700° C. and below the melting-point and cooling quickly or, alternatively, annealing above 700° C. and cooling in diatomaceous earth or in the material known as "Silocel" (Registered Trade Mark). Age-hardening may be effected by quenching in water from above 700° C., followed by heating at 350-550° C. for 15-60 minutes and cooling slowly. See Specification 354,216, International Convention date January 28, 1929, by International Nickel Co., Inc., of New York, assignees of E. M. Wise, of Jersey City, New Jersey, U.S.A.

Cementation

THE carbon content of metals obtained by the thermal decomposition of the corresponding carbonyls is reduced by a heat treatment according to Specification 353,671 (Johnson), dated July 12, 1930, a communication from I. G. Farbenindustrie Akt.-Ges., of Frankfurt-on-Main, Germany. Thus an iron powder so obtained and containing 0.98 per cent. of carbon is sintered at 900° C. in an atmosphere of hydrogen to reduce the carbon content to 0.03 per cent., and a similar iron powder containing 1.3 per cent. of carbon and 1.7 per cent. of oxygen is decarbonised by sintering in a cylindrical mould in a calcining furnace at 1,000° C.

REFRACTORY metals such as tungsten are sintered, in the course of their manufacture, at a temperature not much below the melting point in an atmosphere containing vapour of another metal, e.g., sodium, according to Specification 354,785, dated May 10, 1930, by General Electric Co., Ltd., of London, and C. J. Smithells, of Wembley, Middlesex. A suitable atmosphere is one of hydrogen or of an inert gas such as argon and the sodium, etc., may be vaporised in an adjacent vessel by passing the inert gas over the heated metal before its introduction into the sintering chamber.

Chromium

A METHOD of treating chromium or chromium alloys with gases in the course of their manufacture to improve the quality of the products, is described in Specification 354,387, dated May 10, 1930, by General Electric Co., Ltd., of London, and C. J. Smithells, of Wembley, Middlesex. The metal or alloy is melted in hydrogen which is thereupon replaced by an inert gas, e.g., nitrogen, or, alternatively, the metal or alloy may be cooled in hydrogen and thereafter remelted in nitrogen. The duration of contact of the metal with the inert gas is dependent on its volume, varying, for example, from 15 seconds for a few pounds of metal to more than 1 minute for a few hundredweights.

Concentrating Ores

IN the known process for preparing cassiterite and like ores for concentration by treatment with hydrogen to metallise the surface of the ore particles by reduction, the hydrogen is generated within the pulp by means of zinc alloyed or coated by precipitation with a small proportion, e.g., under 1 per cent., of tin. The concentration may be effected by flotation or by electrical methods dependent on dielectric constants or hysteresis and may be simultaneous with or consequent to the reduction. See Specification 354,395, dated July 24, 1930, by E. J. Lawrence, of Wallington, Surrey, and J. A. Daniel, of London.

Hardening Metal Surfaces

METAL working and like tools are hardened, e.g., in the course of their manufacture, by melting on to the foundation metal a layer of powdered tungsten carbide or other high melting point carbide. The foundation metal may, for example, be heated to redness, whereupon a layer of about 3 mm. of a mixture of tungsten carbide and a flux such as calcium fluoride is applied. A flame of a temperature not below 3,000° C., such as the so-called metallic hydrogen flame, is employed for melting the surface of the foundation metal and the carbide to form a homogeneous layer. Carbides which have been nitrogenised, e.g., by the action of ammonia at 500° C. for about 100 hours, may advantageously be used. See Specification 354,641, International Convention date, November 21, 1929, by F. Krupp Akt.-Ges., of Essen, Germany.

Refining Metals and Alloys

FLUXES for use in refining non-ferrous metals and alloys, especially aluminium and its alloys containing copper or silicon, consist of a potassium compound, preferably the ferrocyanide, ferricyanide or cyanamide, a caustic alkali, a chloride and a fluoride. A suitable flux is, for example, a mixture of potassium ferrocyanide, potassium hydroxide, sodium chloride and calcium fluoride with or without sodium aluminium fluoride. It is preferred to place the flux in a container of the same metal as that to be refined and to introduce this container into the refining crucible and to maintain it therein at as low a level as is practicable. See Specification 353,867, dated March 26, 1930, by F. Dubberley, Senior, F. Dubberley, Junior, C. A. Dubberley, and E. H. Tyson, of Smethwick, Birmingham.

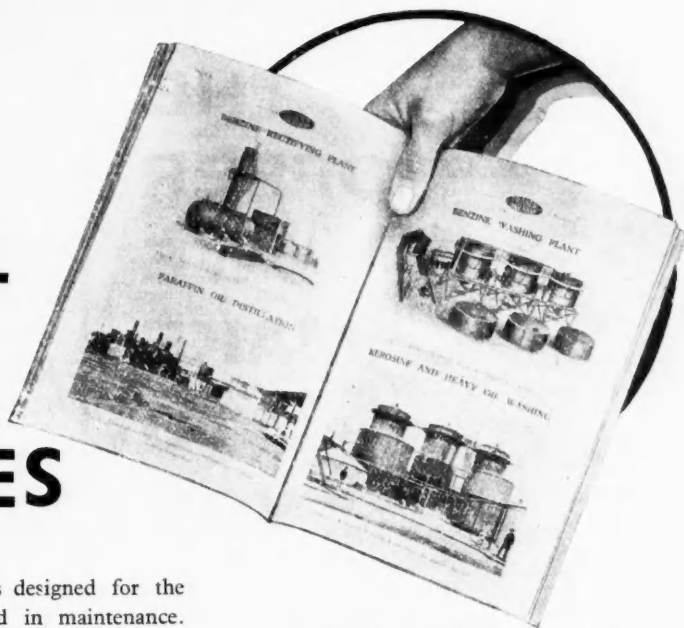
Tungsten

TUNGSTEN-CONTAINING ores, particularly wolframite, are reduced by treatment with a mixture of one or more halogens with several, e.g., 3 to 12 times the volume of hydrogen. Carbonaceous material may be mixed with the ore in cases where the presence of carbon in the product is permissible. Iron and manganese may be removed by volatilisation as chlorides. See Specification 353,815, International Convention date December 31, 1929, by I. G. Farbenindustrie Akt.-Ges., of Frankfurt-on-Main, Germany.

Vanadium

THE reduction, e.g., by carbon monoxide or hydrogen, of vanadium ores, especially such as comprise vanadates of lead or other heavy metals, is effected in the presence of a flux consisting of one or more chlorides or cyanides of the alkali or alkaline earth metals. The heavy metals are thereby obtained in the metallic state and the vanadium as sesquioxide. See Specification 354,816, International Convention date June 24, 1929, by A. Gildemeister, of Paris, and E. Campagne, of Villeurbanne, Rhône, France.

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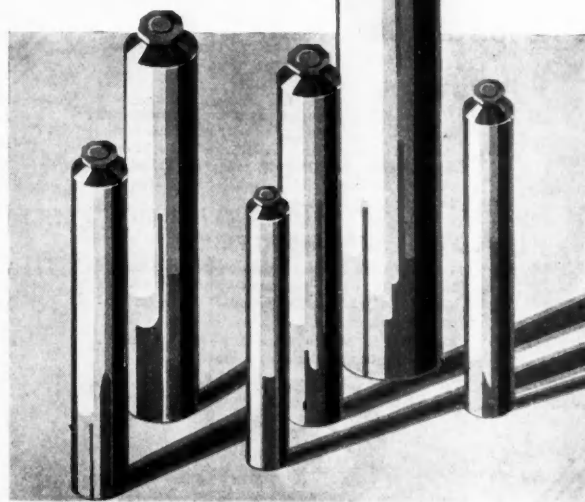
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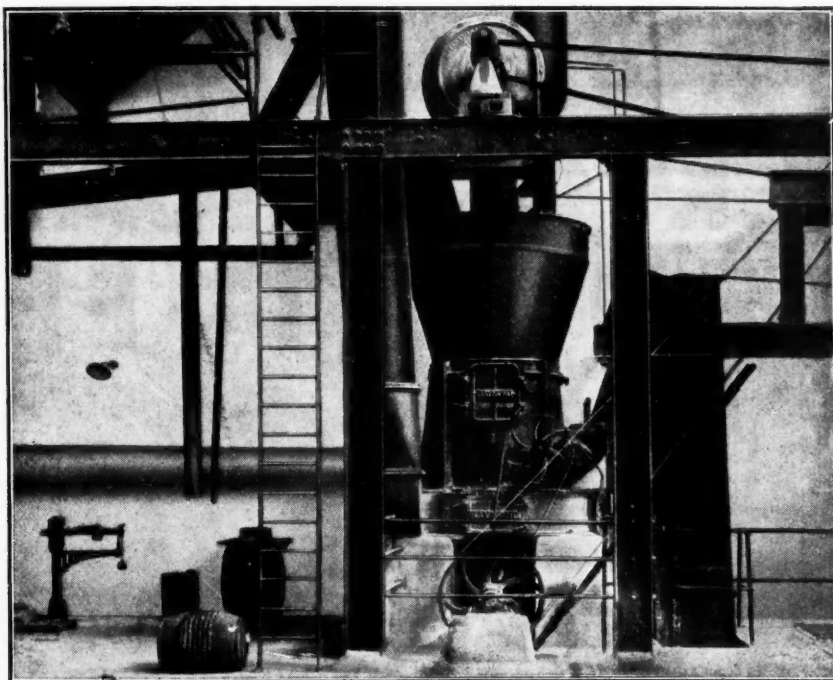
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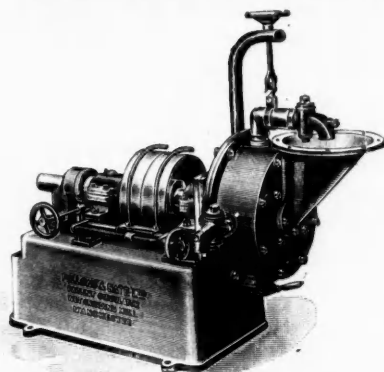
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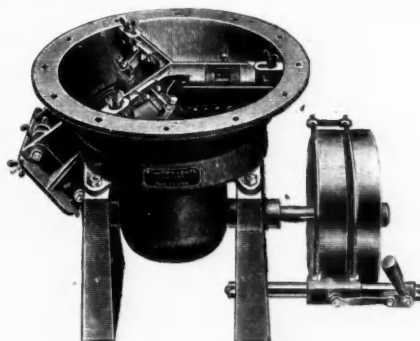
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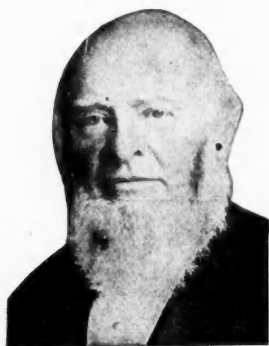
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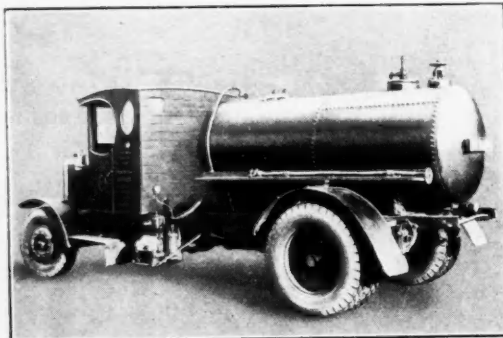
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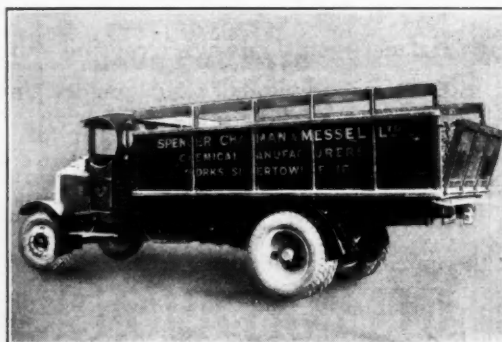
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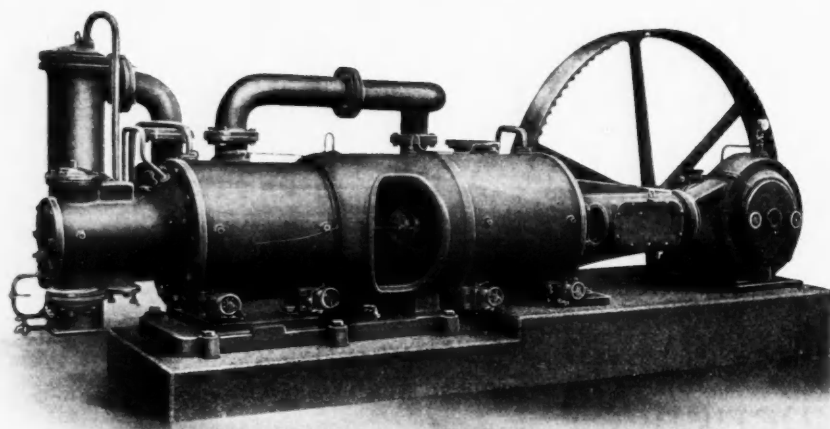
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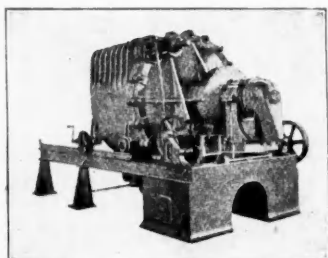
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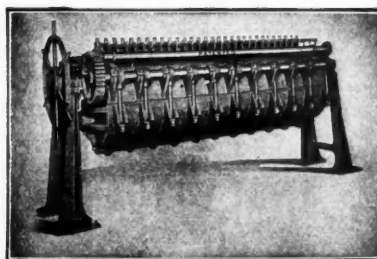
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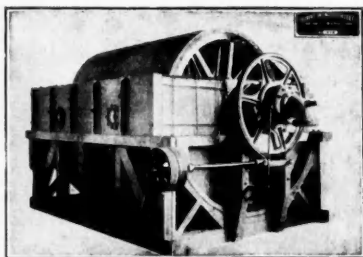


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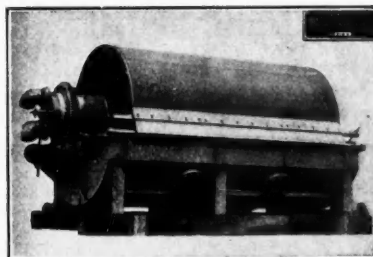
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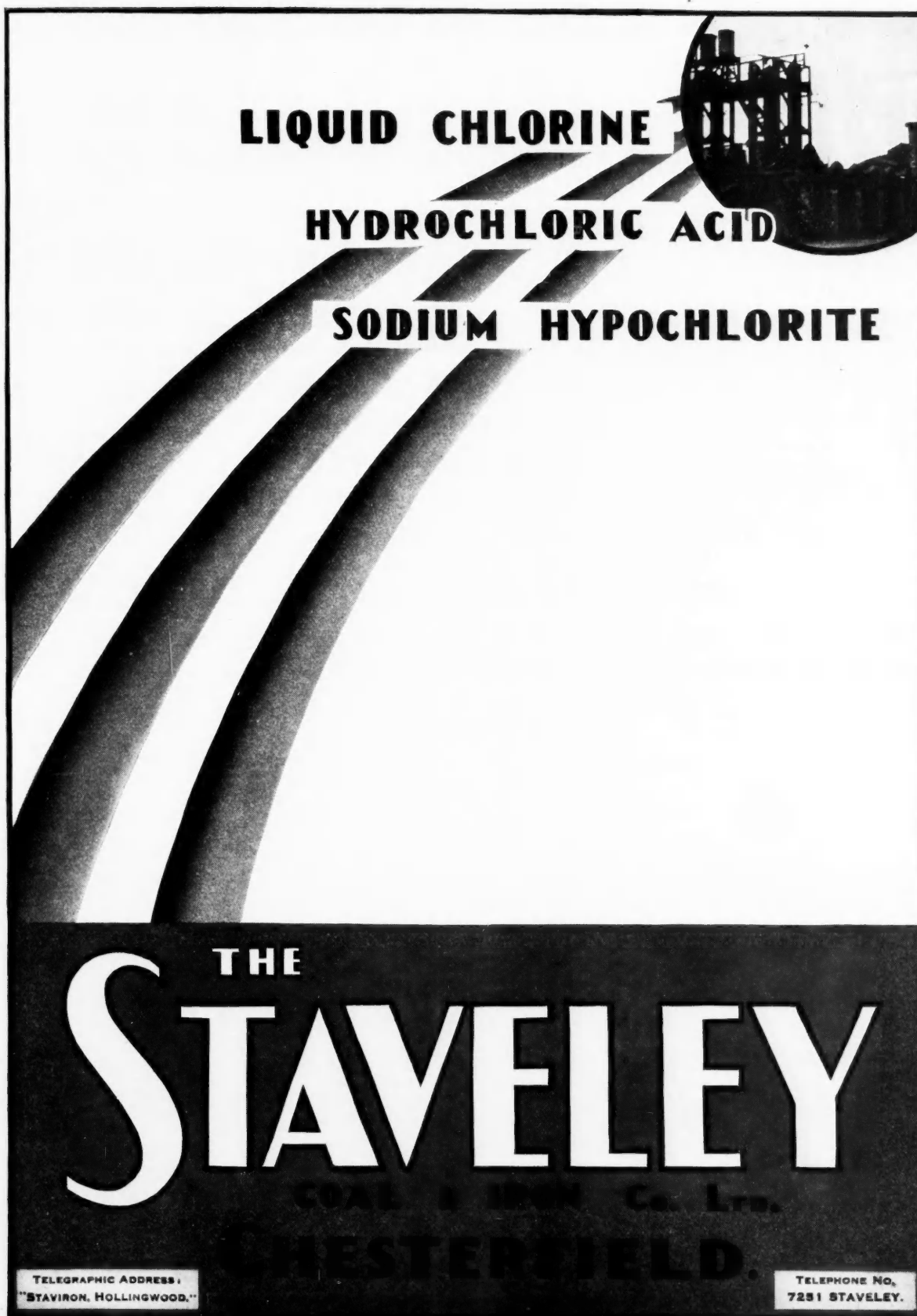
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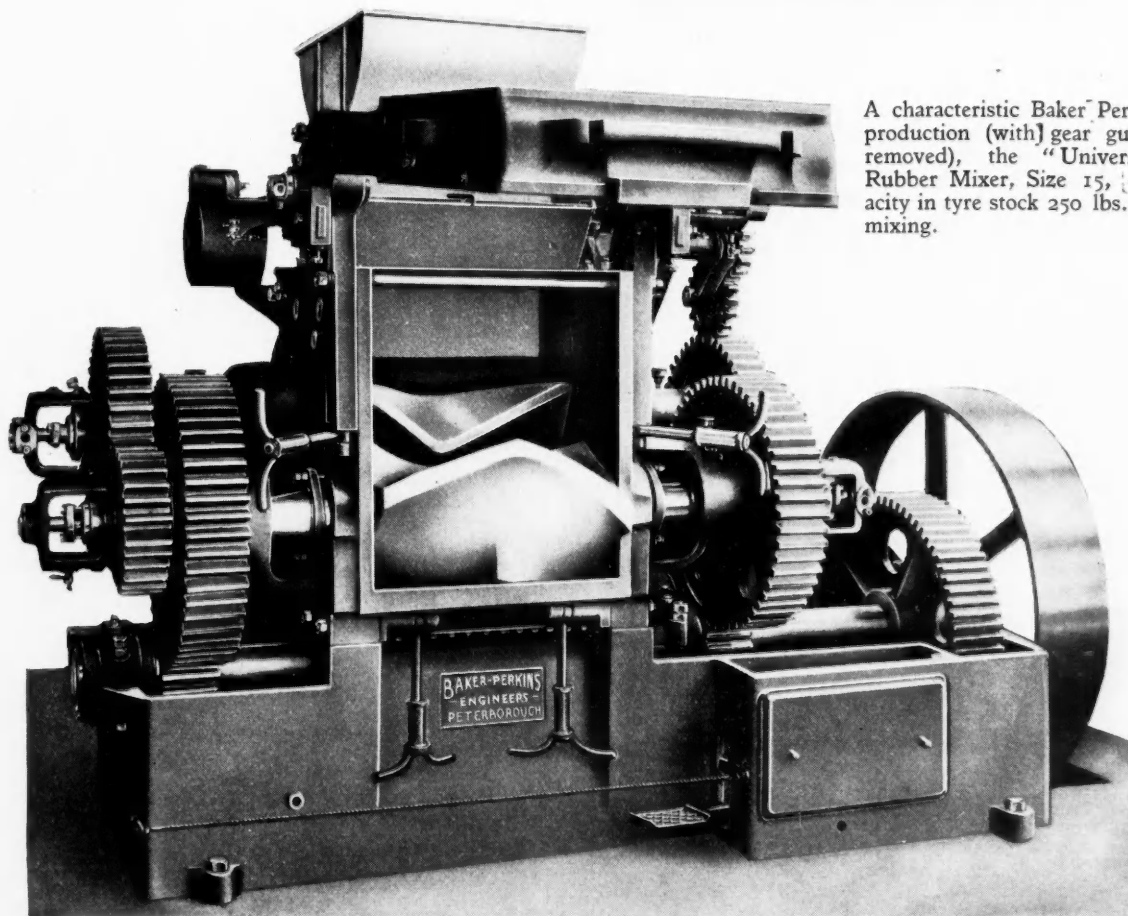
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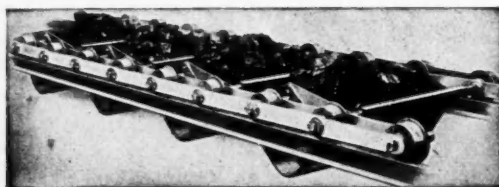
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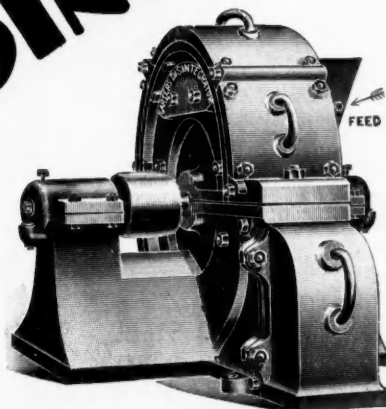
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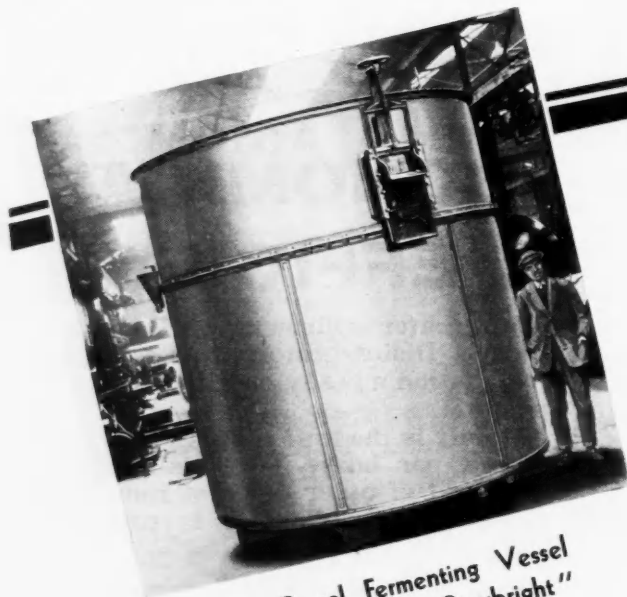
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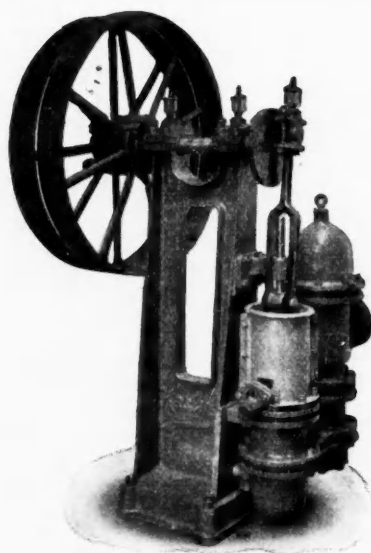
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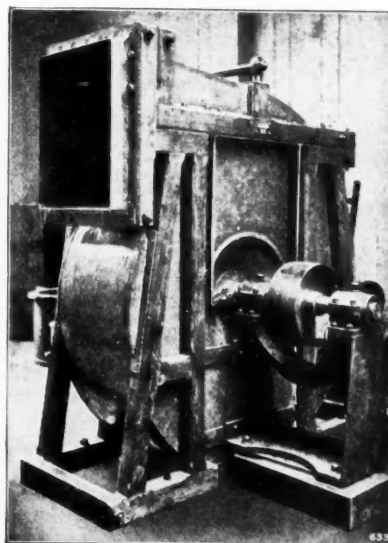


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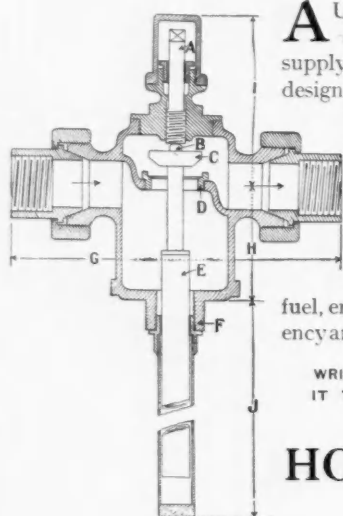
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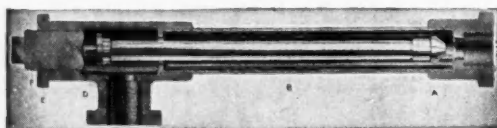


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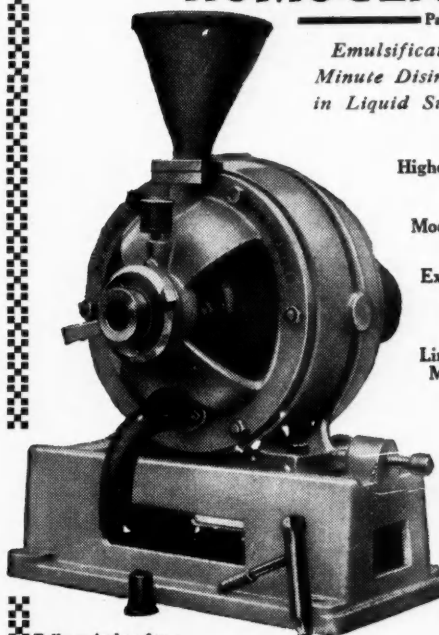
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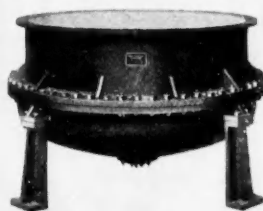
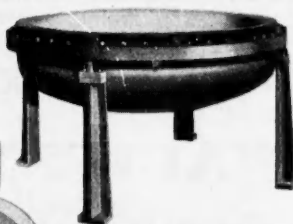
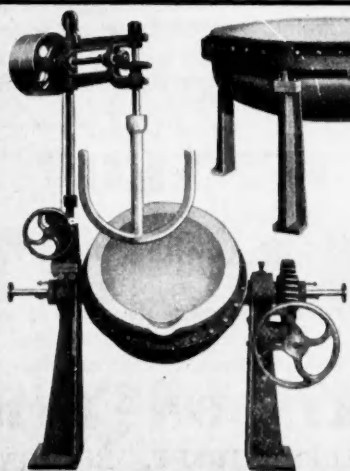


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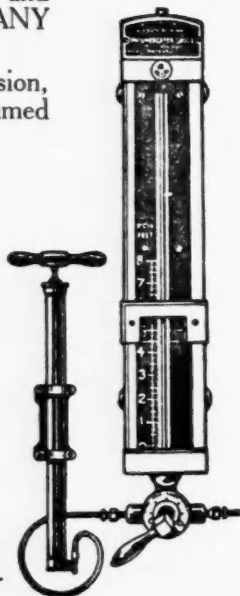
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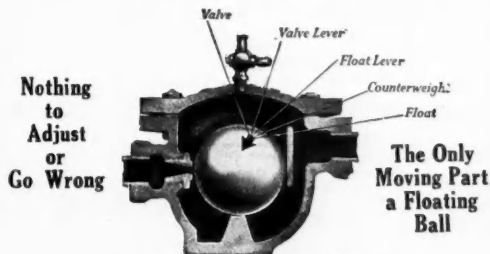
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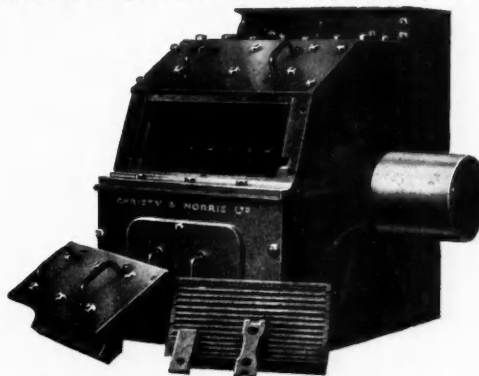
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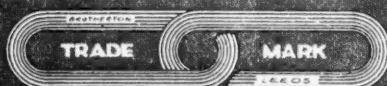
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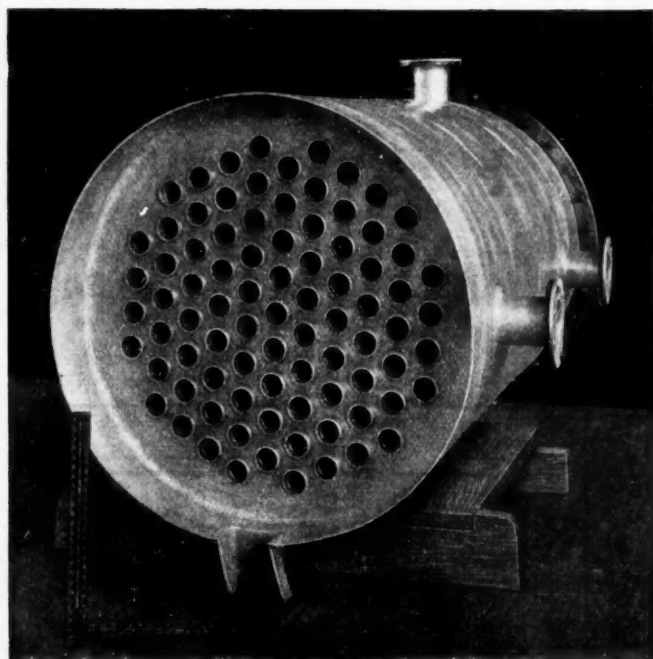
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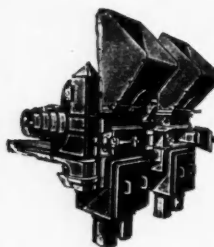
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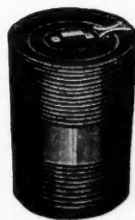
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